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REPORT FOR NASA/MSC
CONTRACT NAS9-8260

EVALUATION OF METAL MATRIX COMPOSITES

REPORT NO. 00.1471
31 AUGUST 1971

SUBMITTED BY

VOUGHT MISSILES AND SPACE COMPANY
LTV AEROSPACE CORPORATION
P. O. BOX 6267
DALLAS, TEXAS 75222

TO

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
MANNED SPACECRAFT CENTER
HOUSTON, TEXAS

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PREPARED BY: K.P. Kelly

REVIEWED BY: B.A. Forcht

APPROVED BY: G.B. Whisenhunt

FOREWORD

This report was prepared by the Vought Missiles and Space Company - Texas of LTV Aerospace Corporation under NASA contract NAS9-8260 Exhibit "C". It was administered under Mr. G. M. Ecord, SMD-Materials Technical Branch as Project Monitor.

This report covers work performed during the period from March through August 1971. The program was performed by the Engineering Materials and Process Group, VMSC-Texas with Mr. B. A. Forcht serving as Technical Manager, Mr. K. P. O'Kelly as Project Engineer and Mr. A. B. Featherston as Fabrication and Test Engineer.

Assistance in the metallurgical studies was provided by Messrs. C. E. Rorick and J. R. Dowell. Mr. R. J. Tufte provided radiator design and thermal analysis.

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1.0 INTRODUCTION

Aerospace requirements for stronger and lighter weight materials capable of operating in increasingly severe environmental conditions have stimulated extensive research in composite materials during the last decade. Federal government sponsored programs have largely advanced the technology in metal matrix reinforced materials to the point where its application to flight hardware is now a reality. A number of studies have shown that the employment of boron/aluminum materials in current and proposed spacecraft can result in substantial component weight savings. Saturn S-II Stage, Minuteman PBCS, the RCS panel of Apollo service module are examples of weight trade studies conducted by NAR. In the Saturn S-II Stage, changing the structural stringer array alone would save 4260 pounds or 45 per cent of the framing weight. Composite skins on the Apollo RCS skin would result in a 36 per cent reduction in skin weight. While the foregoing systems will not be redesigned or retrofitted with composite materials, they represent the potential for composites in future aerospace structures. A space shuttle radiator design represents such a future vehicle application and this report contributes to the data required to employ metal matrix composite materials. The data shows that boron/aluminum is a candidate capable of significant weight savings, at least 30 per cent.

The NASA Manned Spacecraft Center (MSC) is presently conducting a design study of a manned multi-mission space shuttle vehicle. One of the major requirements for the shuttle is that it will be reusable, without extensive refurbishment, for approximately one hundred space missions. Studies have shown that space radiators should be used for orbital heat rejection, but the external surfaces available for radiators must withstand temperatures up to 800°F during reentry (which is above the allowable useful temperature for aluminum - the usual space radiator material). Shuttle designs which require separately deployable radiators stowed inside the cargo bay doors will use deployment mechanisms which are relatively heavy, complex and obstructive to ingress and egress of equipment and personnel through the doors. Externally mounted radiators, made from materials compatible with the thermal environment will eliminate possible leak paths in flexible fluid lines. Externally mounted (low conductivity) titanium radiators may require up to four times as many flow passages to reject the heat load in an external area-limited situation. However, use of a high conductivity material would permit about the same flow passage density of currently baselined designs for aluminum radiators stowed inside the cargo bay.

The extensive development of metal matrix materials over the past ten years has not addressed definition of the thermal properties of the composites (particularly thermal conductivity). The wide variety of material combinations, fiber orientations, and density of fibers selected by the numerous investigators has probably contributed to this lack of baseline data needed for design of a space radiator. The development programs have shown, however, that strength to weight ratios greater than titanium can be obtained at temperatures of about 800°F. The projected high conductivity of aluminum composites makes them strong candidates for radiators. Optimum utilization of the materials, however, requires careful attention to methods of attachment for proper transmission of loads into the composite structure.

The program objective was to determine the feasibility of metal matrix reinforced composites for space radiator designs. Metal matrix composites reinforced with boron or graphite fibers were to be given prime consideration with cusory examinations being made of the influence of the addition of cross plies of stainless steel fibers. Attention was directed toward attainment of fundamental thermal conductivity data for these composites in addition to judging the feasibility of low pressure bonding techniques in order that large size panels, 6 feet x 12 feet might be possible under economic facility conditions. The contractual effort for this program involved the following:

A. Materials Selections
B. Materials Evaluations (in the range of -250 to 800°F)
   1) Tensile strength
   2) Thermal conductivity
   3) Thermal cycling effects
   4) Compression strength at room temperature
   5) Flexural strength
C. Fabricability
D. Non-destructive testing
E. Design analysis
F. Final composite selection

2.0 SUMMARY

This report presents the results of an evaluation of candidate metal-matrix composite materials for shuttle space radiators mounted to external structure. The evaluation was specifically applicable to considerations of the manufacturing and properties of a potential space radiator. Two candidates, Boron/aluminum and Graphite/aluminum were obtained or made in various forms and tested in sufficient depth to allow selection of one of the two for future scale-up programs.

The effort accomplished on this program verified that aluminum reinforced with boron was within the state-of-the-art in industry and possessed properties usable in the external skin areas available for shuttle radiators where re-entry temperatures will not exceed 800°F. It further demonstrated that graphite/aluminum has an apparently attractive future for space applications but requires extensive development prior to scale-up.

Boron/aluminum can be used through the range of -250°F through 800°F for at least 100 flights without degredation in strength and its strength does not fall off even while at 800°F. With a density in the range of 0.093 - 0.095 pounds per cubic inch and better than 100,000 psi tensile strength, its potential is very apparent. For example, titanium which weighs 0.16 pounds per cubic inch loses its strength at 800°F (about 100,000 psi) so that the boron/aluminum would save better than 30 per cent of the weight on hot structures.

The specific objectives of this program were:

(1) Materials Selection - Define requirements for optimum space radiator design by:

(a) Preliminary evaluations of potential fibers including boron and graphite.

(b) Preliminary evaluations of potential aluminum and aluminum alloy matrix metal.

(2) Materials Evaluation - Characterization of the two candidate materials relative to:

(a) Radiator designs including consideration of various fabrication techniques.

(b) Properties of the material consistent with the thermal environment.

(3) Fabricability - attachment processes such as welding and brazing. Secondary and tertiary diffusion bonding methods were emphasized. Normal machining and shaping operations were examined.

(4) Nondestructive tests - methods included radiographic and ultrasonic.

(5) Fabrication - small panels were laid up using secondary and tertiary diffusion bonding techniques.

Because the shuttle requires large areas for radiator surfaces, shuttle radiator hardware requires large-area panel fabrication methods. Additionally, flow passages, structural stiffeners, flow passage penetrations and edge member attachments over a curved surface requires complex fabrication techniques. An approach similar to preimpregnated resin in cloth lay-up used for making reinforced plastics was considered feasible. Metal matrix tapes containing a single layer of filaments can be shaped, spliced and fitted to contour and subsequently laminated and diffusion bonded in an autoclave. Furthermore, this approach affords the opportunity for selectively placed filaments or deletion of filaments in specific areas and thus permits great flexibility in the design.

The test specimens were therefore processed using monolayers of filament in aluminum matrices in such a way as to simulate the foregoing concept. The bonded material thus produced was tested to characterize it within the bounds of an anticipated radiator environment. Having determined that the boron/aluminum specimens were being made in a satisfactory manner, thermophysical properties were determined. Boron/aluminum thermal conductivity ranged from 50 to 80 BTU/Hr Ft °F, making it a far more attractive radiator fin material than titanium at 4 BTU/Hr Ft °F.

Cross-plied boron/aluminum composites have been studied by many investigators since it is seldom possible to design for the high strength uniaxially without some consideration for usable strength in the other directions. The radiator designs presently conceived allow for design of the major loads in the axial direction so that filament orientation can be primarily in that direction. To gain some cross-ply strength, however, a stainless steel cross ply configuration was conceived. Through reference 1 it was learned that stainless steel would afford some increase in cross ply strength with a minimal reduction or compromise in the uniaxial direction. A substantial loss in strength, however was revealed in the tests performed on this program.

Preliminary efforts to produce graphite/aluminum material showed considerable promise. Both nickel coated and aluminum coated fibers could be wetted and bonded into aluminum matrices. These vitally important results encouraged attempts to press bond 5 inch wide tapes. Scale up to larger tapes and laminates revealed that the bonding process will require additional development of processing time and temperature as well as distribution of the filaments.

Significant results of the program included: (1) tensile and flexural strength increases following thermal exposure indicated that the secondary bonding process would probably benefit from a stress relief heat treatment. This increase has been noted in the referenced data but heretofore no clear explanation has been given. (2) Thermal conductivity of boron/aluminum was consistent with predicted values within the constraints of the thermal model. (3) Bonding parameters were shown to be consistent with the low pressure capabilities of the widely available autoclave equipment and in which supplemental heat sources such as blanket heaters can be installed. (4) The mechanical property data was comparable, though scattered, with published data.

Panels made up of boron/aluminum tape were furnished to MSC. The panels demonstrated that secondary and tertiary bonding can be accomplished on complex shaped parts amenable to the fabrication of radiator panel configurations.

3.0 MATERIALS

3.1 COMPOSITE MATERIAL

Metal matrix test material was prepared for this program by secondary bonding of monolayer tapes under vacuum, external pressure and heat. Some material such as the eight ply boron/aluminum and three ply graphite/aluminum were composited by the tape vendor.* Early in the program it became evident that an intermediate material was essential to good bonding and uniformity of filament distribution. Subsequently, a 0.001 silicon/aluminum foil was introduced between each ply and at the surfaces. Consequent to the addition of this "extra" aluminum, the volume ratio of boron was reduced from approximately .50 to .30. An anticipated, and significant, strength reduction was consistent with published data as described in the literature.

In general, the tests were performed by cutting coupons from 10 1/2 sq. inch panels which had been bonded from tape (usually 3 x 3 1/2 x 0.045 inch nominal). The panels were numbered in the order of their fabrication or were additionally designated A and B if two were made one over the other in the same retort. The tests were performed as follows:

Type Test	Type Composite (Aluminum Matrix)	No. of Tests	Test Conditions
Thermal Cycle + Flexural	B/Al	4	-250 to 800°F
	B/Al/StSt	4	-250 to 800°F
Tensile	B/Al	17	-250 to -320, R.T., 400, 800°F
	B/Al/StSt	2	R.T., 400°F
Compressive	B/Al	6	R.T.
	B/Al/StSt	2	R.T.
Flexural	B/Al	16	-250, R.T., 400, 800°F
	B/Al/StSt	10	-250, R.T., 400, 800°F
Thermal Conductivity	B/Al	3	-250 to 800°F
	B/Al/StSt	1	-250 to 800°F
	C/Al	1	-250 to 800°F

* Amercom, Inc., Van Nuys, California

3.1.1 Monolayer Tape, Boron/Aluminum

The monolayer tape used for the evaluations performed during this program was diffusion press bonded from 2 sheets of .002 inch thick 6061-0 aluminum foil having .004 inch diameter boron filaments between foils. The tapes were about 0.005 inch thick made in 5 inch widths x 2 feet long by Amercom, Inc., Van Nuys, California. They were furnished in 45-50 volume per cent and 35-40 volume per cent boron. The filaments were supplied to Amercom by Hamilton Standard (reference 1). The tapes were press bonded into the several configurations by VMSC, LTV Aerospace Corp.

3.1.2 Eight Layer (2, 90° 4, 2) Sheet

The bonded 8 layer sheet used for one thermal conductivity test was diffusion press bonded with 2 outer plies top and botton and 4 center plies of boron oriented 90° to the outer plies. The composite was laid up with 0.002 inch thick 6061 aluminum foil and 0.004 inch diameter boron. The sheet was 45-50 volume per cent boron made by Amercom, Inc. Filaments were furnished to Amercom by Hamilton Standard.

3.1.3 Stainless Steel Tape

The stainless steel tape (rocket wire 0.002 inch diameter AM 350) was diffusion press bonded from 2 sheets of 0.002 inch thick 6061 aluminum foil having the wire sandwiched between foils. The tapes were approximately 6 x 7 x 0.005 inches with approximately 47 volume per cent steel.

3.1.4 Graphite/Aluminum Sheet

Bonded graphite/aluminum sheet used for the evaluations was press bonded by Amercom using Morganite high modulus graphite tow 0.0002 inch diameter. The tow had been treated by VMSC and furnished to Amercom. Exploratory development of the process of treating of the graphite filaments and bonding of the tow to aluminum was performed by VMSC followed by pressing techniques developed by Amercom.

3.2 PROCESSING MATERIALS

3.2.1 Stop-Off Materials

Diffusion bonding techniques always include ways to either prevent metal surfaces of the tooling materials from bonding together or removing bonded tooling, subsequent to the pressing operation, by chemical or mechanical means. Prevention by use of stop-off material application is a relatively simple procedure unless operations call for bonding temperatures near the melting point of sample material. Cutting away the tooling material would be impractical for sheet composites. The use of temperatures high enough to soften aluminum resulted in diffusion bonding to the glide sheet. Solution to the problem required experimentation. Published information and commercial stop-off materials proved to be inadequate. A series of trial runs were conducted to evaluate both known and new materials which resulted in the selection of a "mist" sprayed solution developed under another program. Best results were obtained when a very thin coat of this material was fogged on an oxidized Rene '41 glide sheet. Coating the glide sheet effectively prevented sticking to the specimen. A smooth, silky finish was obtained on the surface of panels subsequently processed for mechanical and physical property tests.

3.2.2 Processing Adhesive

It was quickly apparent that assembly of a number of small pieces of tape, separator sheet and pressure plates would require a means for maintaining orientation of all parts until bonding pressure was applied. A type of non-residue adhesive is generally used by fabricators of metal matrix composites to maintain alignment of filaments during lay up and press bonding, see reference 2. Three types were evaluated and one was selected. This was solid, pure polystyrene dissolved in toluene. When heated under vacuum and outgassed at 450, then 650 and then 800°F, all traces of residue were removed. The other two materials would not completely volatilize but left a slight smut remaining inside the retort. The adhesive served a second, but vitally important function in that it protected the cleaned tape surfaces while being assembled, baked out and installed in the retort. Upon assembly, all tape components were dipped in a thin solution of the adhesive immediately following cleaning operations, thus eliminating the need for special storage and handling procedures.

3.2.3 Intermediate Materials

In order to accomplish bonding without degrading the strength of the final composite structure it was essential to avoid filament-matrix reactions caused by exposure to excessive time at temperature. This is commonly accomplished by placing a lower temperature diffusion media at the bond interface. Early attempts at selecting the intermediate material led to trial runs with (1) 13 per cent silicon/aluminum powder spread between plies, (2) copper plated tape surfaces and (3) a 7 1/2 per cent silicon/aluminum foil 0.001 inch thick. The powder failed to flow uniformly and resulted in numerous unbond areas and lumps of unfused powder. The copper formed a brittle intermetallic compound but the 7 1/2 per cent Si/Al produced a consistently good bond and permitted an acceptable time-temperature exposure without the attendent embrittlement problems. The Si/Al foil was therefore selected as the intermediate material to be used for all secondary and tertiary bonding operations.

It has been shown that the matrix plays an important role in the behavior of the composite in its formulative stages. Copper added to the matrix, either in the starting material (2024) or as a plating is detrimental to the final composite. The rationale is that the eutectic which forms above 548°C leads to gross intermetallic embrittlement because slow cooling from the bonding temperatures allows the copper to precipitate rather than remain frozen in solid solution (reference 3). It is this embrittling effect which normally prohibits fusion welding or brazing of these alloys. A cursory evaluation of copper as an intermediate material was performed as part of the effort to examine all material systems which have been used by other investigators to aid in the bonding of aluminum composites. As suspected, metallurgical examinations as well as poor bonding results verified that the embrittlement can occur.

3.3 PROCESS TOOLING MATERIALS

Retorts were made from Rene '41 and hard rolled Type 321, annealed Type 321 and PH 15-7 stainless steel. They were both brazed and welded. Brazed retorts made from 321 and PH 15-7 proved to be acceptable from the standpoint of ease of forming, joining, handling and sealing. Rene '41 separator (glide sheet) was selected originally because of high temperature stability and a tightly adhering oxide surface appeared to provide some stop-off advantages.

Early runs using thin (1/8 inch or less) stainless steel pressure plates resulted in non-uniform pressure. An increase to 1/2 inch thick plates proved suitable and mild steel was used because of availability. Figure 1 and 2 illustrate the process and process tooling.

4.0 EQUIPMENT AND PROCEDURES

4.1 EQUIPMENT

Presses consisted of Carver Laboratory Model B presses having special heated plattens. Temperature of the plattens and retort were monitored and recorded with Honeywell controllers and recorders. A Duo Seal laboratory vacuum pump, rated at 10^{-4} mm Hg was used to evacuate the retorts. Dry nitrogen was connected into the evacuation system to prevent loss of specimen material in the event of a vacuum line or retort leak.

Conventional Tinius Olsen tensile test equipment was used as illustrated in Figures 3 and 4 to perform flexural, compression and tensile tests at the test temperatures required. These machines were equipped with temperature controlled chambers.

Thermal conductivity apparatus (Axial Rod) was located at Dynatech Corporation of Cambridge, Massachusetts where all thermal conductivity determinations were made. (See Figure 5)

4.2 PROCEDURES

4.2.1 Panels Fabrication

Initially, the fabrication of test material was made by laying up laminates of tape material cut to the dimensions of the various test specimens in the interest of conserving material and avoiding damage incurred by cutting operations. Stacked strips were laid side by side in order to make several specimens simultaneously. Very difficult handling of the many small strips precluded continuation of this practice. Panels were subsequently fabricated from which the specimens were rough cut on a diamond wheel and smoothed on a diamond grinder. A discussion of the materials fabricated and processes used was presented in 3.0.

4.2.2 Testing

Mechanical property testing was performed in accordance with tentatively established methods developed for metal-matrix materials and Federal Std. 151. The procedures and specimen configurations (Figure 6) differ somewhat from those used for conventional materials. Flexural testing followed the ASTM proposed method (reference 4). Elevated temperature flexural and tensile tests were run totally enclosed in chambers. Low temperature tensile tests were run with liquid nitrogen surrounding the test section of the specimens.

Thermal cycling exposure of flexural specimens was performed by alternately placing the specimens in a heated oven and suspending them over liquid nitrogen. A dummy specimen having a buried thermocouple was used to monitor and adjust the rates of heating and cooling. The rates were based on a possible reentry profile of the shuttle as indicated by Figure 7.

Thermal properties of potential composite materials for use in space radiators was the primary objective of this program. As shown in Table I the thermal properties and weight effectiveness of the aluminum composites is potentially good. Mechanical properties were determined and inspection investigations were performed to make sure the thermal property measurements were being determined on representative material. Having satisfied the requirements to test a nominally good material, prime consideration was given to processes amenable to large panel fabrication.

5.0 RESULTS

Results of the testing on this program were expected to verify the relationships of various candidate radiator materials as shown in Table I where the aluminum composites offer a very substantial weight advantage. Specifically, this report presents the following properties of boron/aluminum and boron/aluminum containing stainless steel cross plies.

5.1 TENSILE

Table II and Figure 10 present the influence of shuttle re-entry temperature exposure upon tensile strength. Through the full excursion from -250°F (or lower) to 800°F, a radiator could withstand high stresses parallel to the filaments. However, the addition of stainless steel cross plies severely degraded tensile strength.

5.2 COMPRESSION

Table III and Figure 11 show that the compression strength of unidirectional boron/aluminum is far superior parallel to the filaments as opposed to cross ply strength. The addition of steel cross plies severely reduces the compressive strength.

5.3 FLEXURAL

Tables IV and V and Figure 12 illustrate that with the boron filaments all in the parallel direction the flexural strength can be expected to exceed 100,000 psi through 800°F. Figure 12 shows that cross ply strength was not improved by incorporating stainless steel. Additionally, Table VI and Figure 13 show that repeated (100) re-entry like cycles do not degrade the material.

5.4 FLEXURAL MODULUS

Table VII and Figure 14 illustrate that parallel to boron filaments, the modulus of B/Al is superior to titanium at room temperature (16×10^6 psi) through 400°F.

5.5 THERMAL CONDUCTIVITY

Figures 15 through 19 present the results of thermal conductance testing. Detailed discussions of the data are presented in section 6.5. The curves provide the filament orientation relationship for design and show that the highest conductivity is parallel to unidirectional filaments. The graphite/aluminum curve shown in Figure 15 is presented but should be regarded as inadequate since good bonding was not achieved.

5.6 COMPARISON WITH OTHER INVESTIGATORS DATA

Using current fabrication techniques, the generally accepted filament volume per cent in boron/aluminum composites is 50. Strength of these composites is commonly reported to be about 180,000 psi UTS. Attempts to use higher filament ratios have resulted in broken fibers, porosity and poor filament distribution in the matrix. In this work, 50 V/O tapes could not be bonded together directly because (1) there was insufficient matrix material to allow the filaments to remain equally spaced and unbroken under the bonding pressure and (2) the secondary bonding temperature was high enough to cause degrading boron reaction with the matrix. An investigation of these problems and consultations (per reference 1) led to the concept of adding an intermediate material in foil form. This added sufficient matrix volume to allow the filaments to remain undisturbed plus lowering the required bonding temperature. A very thin film of intermediate material less than 0.0005 inches thick would have accomplished these purposes but availability of suitable materials in such thin films is limited. The only readily available intermediate material which could serve both purposes was a 0.001 inch thick alloy of aluminum containing 7.5 per cent silicon. This meant that there would be a substantial increase in matrix volume; in the order of 35:65 boron to aluminum ratio. Comparing this volume ratio the mechanical properties obtained were within the average of other reported data as illustrated in Figure 20 (see reference 5). For space radiator applications, the proper selection of volume ratios should result from a trade off of strength and thermal conductivity.

5.7 NON-DESTRUCTIVE TESTING

Ultrasonic and radiographic inspection methods were investigated as non-destructive test methods to supplement visual inspection on both the tape and the diffusion bonded laminates. Figure 21 presents a radiograph of a satisfactory panel whereas a faulty splice may be seen in the radiograph in Figure 22. Ultrasonic inspection was able to find defects (unbonded areas) on the edges of panels but was not effective in locating small internal defects. The "C" scan recording in Figure 23 shows sound material with some disbond in the corners.

Ultrasonic inspection of the tape was very effective in locating defects. Figure 24 is a "C" scan recording of a well bonded foil while Figure 25 shows a similar tape with large disbond areas. The graphite foil which appeared to be of questionable quality (Figure 26) was ultrasonic and radiographically inspected. Figure 27, "C" scan recording shows large areas of disbond and the radiograph (Figure 28) shows bunching of the graphite fibers, a condition which would prevent good diffusion bonding.

Both radiographic and ultrasonic inspection techniques exhibit good applicability to basic tape and laminated panels examination. The analysis of the results obtained by either is enhanced by comparison with results obtained with the other method. In combination, high quality inspection requirements could be developed for both the tapes and diffusion bonded panels.

6.0 DISCUSSION OF RESULTS

6.1 TIME-TEMPERATURE EFFECTS

The most significant process parameter noted during the progress of this program was the time - temperature profile used to accomplish a suitable secondary or tertiary diffusion bond. It became apparent that the profile was not necessarily coincident with that used by the manufacturers of the original tape or starting material particularly with regard to maximum temperatures. Additionally it appeared that the pressure can be substantially reduced in secondary and tertiary bonding since it is not necessary to force the aluminum around and in between filaments. Thus the low pressure, autoclave-like process possibility becomes an attractive approach to simplified production practices.

6.2 FILAMENT-MATRIX INTERACTIONS

The amount of diffusion and reaction that occurs between the matrix and the filament strongly affects the material properties and the mechanical and chemical behavior of the composite. The reaction of the boron with aluminum is illustrated in Figure 29. Similarly, Figure 30 shows the interaction with stainless steel resulting from excessive time and/or temperature. The diffusion increases with increase in the time - temperature parameter. The condition had not been observed in any other sample and an investigation showed that the stainless steel tape contained some disbonding as well as small areas of reacted filaments. Reference 6 discusses some of the studies that have examined the problem of interfacial reactions.

The diffusion of nickel coated graphite was evidenced dramatically in metallographic study where the nickel exhibited considerable mobility and can be seen as white areas displaced in the matrix rather than surrounding the filaments. This displacement is considered a possible contributing factor in the failure to achieve successful secondary bonding, the graphite filaments having been, in effect, become uncoated.

6.3 THERMAL CYCLING STABILITY

Flexural specimens were tested at room temperature after exposure to the thermal excursion which was scheduled to simulate that of a possible shuttle radiator (Figure 7). Thermal stability characteristics were evaluated on a 7 ply unidirectional lay-up configuration of the boron aluminum and a 6 ply unidirectional boron + 2 middle 90° cross plies of stainless steel (AM350 rocket wire).

Figure 13 illustrates that the exposure to 100 cycles of -250 to 800°F had no degrading effect of the flexural properties at room temperature and no metallurgical degradation could be observed. Typical post thermal cycling microstructures are shown in Figure 31, but no effect of thermal cycling is seen in the grain structure. The apparent increase in strength in the direction of the filaments may be the result of matrix stress relief. The increase in properties was consistent with all tests run on specimens parallel to the filament where the material was subject to a thermal environment. These increases have been noted by other investigators but no positive explanation has been offered. Certainly, a specific reason for the increases could lead to improvements through thermal treatments and an optimum thermal process may be profitable.

6.4 GRAPHITE/ALUMINUM

Graphite filaments in aluminum is such a new material that there are many questions open regarding compatibility at both processing and service temperatures. Early testing has shown that the graphite filament will react and degrade at elevated temperatures but that barrier coatings tend to prevent degradation. Nickel is the most commonly used barrier. The disadvantages of using nickel in an aluminum composite include a density increase and poorer filament distribution. Also, the nickel has a tendency to migrate toward the center of the bundle of filaments.

Externally mounted radiator design criteria indicate the desirability of high stiffness as well as high conductivity materials of construction. The boron filament in the conventional boron/aluminum composite has low thermal conductivity whereas graphite filament conductivity should be higher. It is much more readily machined than boron and tapes can be formed over smaller radii. The inducement to exploit high modulus graphite reinforcements then is obvious. Hampering industries efforts to make sound graphite/aluminum composites has been wettability of graphite by aluminum.

As shown in Figure 32, good metal wetting of graphite filaments was accomplished at VMSC using a sensitizing treatment followed by an electroless nickel coating process. With these filaments VMSC was successful in diffusion bonding 1/2 inch samples. Amercom supplied samples of aluminum coated (electroplated) filaments as shown in Figures 33 and 34. As described in their reports (reference 7) Aerospace Corporation, Los Angeles has shown that aluminum can be infiltrated into small bundles of graphite filaments. Thus, wettability appears to no longer be an obstacle in the way of good graphite/aluminum composites. However, the material shown in Figures 26, 27 and 28 illustrates the existence of scale-up problems and practical fabricability of large sheets and panels requires extensive laboratory and manufacturing development.

6.5 THERMAL CONDUCTIVITY

Extensive mechanical property data exists for boron/aluminum but thermophysical data is sparse in the literature. No good data of any kind exists for graphite/aluminum. In Reference 8, thermal expansion was determined for a 35 V/O boron/aluminum which was in good agreement with predicted values based on rule of mixtures of the constituents. This data will be useful in radiator design. Thermal conductivity, on the other hand, had not been measured.

The thermal conductivity is a critical material property needed for utilization of a material in spacecraft thermal control systems and is particularly important for space radiator design. Figures 15 through 19 show data obtained in environment testing by Dynatech using a standard thermal conductivity measuring apparatus illustrated in Figure 5. Samples were uniformally prepared as shown in Figure 34. Because of the great difference in the thermal conductivity of the aluminum matrix and the boron filament, it is expected that thermal conductivity should be quite sensitive to details of the fiber arrangement. For conductance parallel to fibers it is expected that thermal conductivity should follow approximately a rule of mixtures of the material. For conductance across fibers, however, heat flow through the aluminum must follow a torturous path around the boron fibers and it is not intuitively apparent what kind of model should be used to predict composite thermal conductivity. An additional problem is the unknown thermal conductivity of the boron fibers, but, since it is expected to be significantly less than aluminum, the aluminum heat transfer should dominate the heat flow process. The aluminum matrix conductivity, however, is not precisely known because of the mixture of alloys used in the tape and bonding processes. The following paragraphs (a) define models used to predict upper and lower limits on thermal conductivity for the composite material (b) compare predicted properties to test data and (c) define a recommended model for predicting conductivity of a composite layup.

6.5.1 Analytical Models

Two analytical models are derived: (1) one for the upper limit on conductivity (K_u) based on the rule of mixtures and (2) one for the lower limit on conductivity (K_l) based on an intuitive model of the effect of cross plies. Thermal conductivity K is defined by $Q = KA \frac{dT}{dx}$ where Q is the heat transfer through area (A) and $\frac{dT}{dx}$ is the change in temperature along the heat flow path. Assuming a uniform temperature distribution in all components of a composite sample the composite conductivity K_c of the total cross sectional area A_T can be expressed as:

$$K_c A_t = \frac{Q}{dT/dx} = K_1 A_1 + K_2 A_2 \ldots K_N A_N$$

where A_1, $A_2 \ldots A_N$ are the effective conductance areas of the constituent (1, 2, ...N).

An upper limit on K_c (K_u) can be estimated by assuming that all fibers act as if they were oriented with their axis parallel to the heat flow path so that:

$\frac{A_N}{A_T} = \frac{V_N}{V_T}$ where $\frac{V_N}{V_T}$ is the volume ratio of the Nth component.

$$K_u = K_1 \frac{V_1}{V_T} + K_2 \frac{V_2}{V_T} + \ldots K_N \frac{V_N}{V_T}$$

For a lower limit on conductivity it is assumed for cross plies that the torturous path through the aluminum results in a net effect of the fibers blocking heat flow as if they formed flat sheets with a dimension t_n equal to the diameter of the fiber. Figure 36 illustrates the model used for K_L. t_T is the total thickness of the specimen and t_X is the sum of the diameters of the cross ply fibers. The analytical model is then:

$$K_L = K_a \frac{V_a}{V_T} \frac{(t_T - t_x)}{t_T} + K_b \frac{V_b}{V_T} \frac{(t_T - t_x)}{t_T} + K_X \frac{t_x}{t_T}$$

This model makes the simplifying assumption that the volume ratio of the longitudinal fibers is applicable to the total specimen cross section. Since there is some variability in volume ratio along the specimen length, the volume ratio is only an estimate, and the complexity introduced by refinement of the analyses is not justified. The equation for K_L is therefore somewhat conservative and representative of a lower limit since consideration of the effect would tend to increase the effective conductance.

6.5.2 Prediction of Test Results

The analytical method will be illustrated for sample number 4 (the most complex specimen); the results for the four samples are tabulated in Table IX and illustrated in Figure 37. The following room temperature thermal conductivities (BTU/hr, ft°F) are assumed for the conductivity of aluminum (a) boron (b) and stainless steel (x):

$$K_a = 107 \frac{\text{BTU}}{\text{hr ft}^\circ\text{F}}, \quad K_b{}^{*} = 20.9 \frac{\text{BTU}}{\text{hr ft}^\circ\text{F}}, \quad K_x = 9. \frac{\text{BTU}}{\text{hr ft}}{}^\circ\text{F}$$

The analysis uses the following dimensions and volume ratios:

(1) diameter of cross ply fibers = 0.002 in.

(2) volume ratio of stainless cross ply region is 47% stainless in aluminum matrix

(3) volume ratio of boron in boron region is 35% boron in aluminum matrix

(4) $t_T = 0.047$

(5) thickness of cross ply region = .0087

$t_x = 2\ (.002) = 0.004$

$$\frac{t_T - t_x}{t_T} = \frac{.047 - .004}{.047} = .915$$

* (based on Reference 9 Purdue Thermo physical properties for polycrystalline boron)

$$\frac{V_b}{V_T} = 0.35 \quad \frac{0.047 - 0.0087}{0.047} \quad = .285$$

$$\frac{V_x}{V_T} = 0.47 \quad \frac{0.0087}{0.047} = .087$$

$$\frac{V_a}{V_T} = 1 - \frac{V_b}{V_T} - \frac{V_x}{V_T} \quad = .628$$

$$K_u = K_a \frac{V_a}{V_T} + K_b \frac{V_b}{V_T} + F_x \frac{U_X}{V_T}$$

$$= 107(.628) + 20.9 \ (285) + 9\,(..087)$$

$$= 67.1 + 5.96 + .782$$

$$K_u = 73.8 \text{ BTU/hr ft}^\circ\text{F}$$

$$K_L = K_a \frac{V_a}{V_T} \quad \frac{t_T - t_X}{t_T} \quad + K_b \frac{V_b}{V_T} \ \frac{t_T - t_X}{t_X} + K_X \frac{t_X}{t_T}$$

$$= 107(.628)\ (.915) + 20.9(.285)\ (.915) + 9 \ \frac{.004}{.047}$$

$$= 61.5 + 5.45 + .78$$

$$= 67.7 \text{ BTU/hr ft}^\circ\text{F}$$

6.5.3 Conclusions About Thermal Conductivity

A review of the analytical technique reveals that the predicted thermal conductivity is quite sensitive to the aluminum volume ratio and the value used for aluminum thermal conductivity since this dominates the heat transfer. For the analytical models the lower limits of aluminum volume ratio and a value of 107 BTU/hr ft°F for the conductivity of the aluminum at room temperature yielded results that correlate well with test data. This is also a reasonable value to use for the aluminum in the composite material which is approximately 80% 6061 aluminum and 20% of a 7-1/2% silicone aluminum alloy.

Inspection of Figure 37 reveals that the analytical models yield a very broad range between K_U and K_L when the cross plies have a significant or dominant effect (Samples 1 and 2). For materials of this type the resulting K_C appears to fall between K_L and $\frac{K_U + K_L}{2}$. For samples where cross ply effects do not dominate (Samples 3 and 4) K_C more closely approaches the values predicted by K_U.

It is concluded that the analytical models developed can be used to provide a reasonable estimate of a composite material thermal conductivity, and it is recommended that their use be investigated for a broader range of temperature and material compositions.

6.6 EFFECT OF TEMPERATURE ON MECHANICAL PROPERTIES

The tensile, flexural and compression results at ambient, cryogenic and elevated temperatures are presented in Tables II through

VII and shown in Figures 10 through 14. These values characterize the composites to be of reasonably good quality in light of the present state-of-the-art in B/Al. Although the tests conducted herein were conducted with high pressure bonding processes as illustrated in Figure 38) preliminary tests have shown that an equally good bond can be obtained at low pressure (see Figure 39). A review of the numerous publications in reference 11 and 12 will reveal the various approaches investigators have taken to the prediction of properties of composites. A specification prepared by General Dynamics as shown in Reference 4, requires 115,000 psi longitudinal, 12,000 psi transverse tensile strength and 24×10^6 psi modulus. The effects of exposure to the thermal cycles predicted for shuttle radiators were not entirely unexpected based on the literature referenced. The exposure alternating from cryogenic to 800°F, however, had not been conducted and therefore it was considered desirable to check this excursion. Other investigators including Reference 10, have found that in time the material suffers severe degradation. Krieder found a drastic loss in strength after 500 hours at 200°C (Reference 5). Thermal cycling (-250 to 800°F) 100 times had no apparent effect on the flexural strength of the boron/aluminum, and in fact appeared to increase the mechanical properties as already discussed. The addition of 2 cross-plies of stainless steel rocket wire had the effect of reducing material properties significantly but there was still an increase in strength due to the thermal cycling.

6.7 ANISOTROPY OF THE COMPOSITE

The well known anisotropy of fibrous reinforced materials was examined in view of potential radiator designs where the major loads can be readily oriented. For unidirectional boron/aluminum the transverse properties are as low as 10 per cent of the longitudinal properties, however, to improve the ratio, 2 cross-plies of 300,000 psi stainless steel (AM 350) were considered potentially effective in increasing transverse properties without serious compromise from longitudinal properties of the unidirectional lay-up. The concurrent degradation of longitudinal strength can be partially attributed to the effective reduction of volume per cent of boron (15 per cent). Additionally, the stress concentration effect of transversely oriented filaments is substantial though not readily determinant.

6.8 MANUFACTURING

6.8.1 Low Pressure Bonding

In view of the potentially large panels needed for space radiators, manufacturing techniques were examined which will lead to economical production of large parts. A most desirable process incorporates the use of large size autoclave equipment. Although autoclaves do exist which are capable of high pressures, the larger and more common autoclaves operate at 200 psi. Thus, it is advantageous to show feasibility of diffusion bonding composite tapes, laying them up and bonding an assembly in a similar fashion to fiberglass lay-ups. Preliminary tests show that diffusion bonding of composite tapes can be accomplished at 200 psi. The tests showed flexural strengths in excess of that achieved at 2,000 psi.

6.8.2 Splicing Potential

The possibility of splicing sheets together in order to manufacture large panels with tape of smaller dimension than desired was examined. Five half inch wide assemblies of 7 plies were laid up side by side and bonded into a 2 1/2 inch wide sheet. Radiographic (Figure 21) and "C" scan ultrasonic examination showed that the joints were well healed. However, a second specimen with tape laid in alternate layers as seen in Figure 22 showed a tendency for the tape to shift and lap over adjacent tape. Tape spliced parallel to filaments is apparently feasible so long as care is taken to prevent adjacent tapes from shifting. Obviously, tape butted together perpendicular to the filament direction also seen in Figure 22 would require overlapping or staggering of the tapes.

6.8.3 Other Joining Processes

Reference 4 describes, comprehensively, the welding and other joining work which has been performed and current programs include joining processes. This program examined this effort and performed minimal welding, brazing and soldering operations for a first-hand look at the problems. Spot welding of boron aluminum results in excellent shear strengths and is higher than for equivalent thicknesses of conventional aluminum. Extensive tip pick-up and spitting were noted, however, indicating manufacturing techniques are not as straight forward as standard spot welding. Attempts to fusion weld quickly pointed up the effect of melting the two different materials (boron and aluminum) simultaneously. Generally it appeared that the high resistance of the boron caused the filaments to burn away before the aluminum could melt to form a good matrix fusion zone. Lead-tin soldering and aluminum brazing were accomplished readily. A zincate coating or a nickel coating on the surface of the aluminum was necessary, as with conventional aluminum, prior to lead-tin soldering.

Diffusion bonding with both high and low pressure processes (Figures 38 and 39) was used to fabricate shapes as shown in Figure 40. The ability to form the composite as shown in the photograph hinges on the fact that monolayer tape is flexible enough to bend over a 3/8 inch radius without breaking fibers. Once the tapes are bonded, very little can be done to form over radii. The fabricated panel, then, shows that a complex shape is possible where strength requirements necessitate filaments parallel to the hoop direction of a curved element. Secondly, a tertiary or third order bonding process to join two complex shaped elements is demonstrated since the top curved piece and bottom flat piece were joined by bonding in a second operation at VMSC. It is expected that a space radiator design could incorporate an extruded aluminum flow passage sandwiched between two pre-formed (during secondary bonding) panel elements and bonded into position in a tertiary bond operation. Since the flow passages are not parallel but change direction over the panel surface, splicing, as described in 6.8, would become necessary. This would be accomplished by stragetic splicing of individual tapes in the lay-up.

7.0 CONCLUSIONS

The conclusions which can be drawn from this program are:

(1) Boron/aluminum thermal test results showed this material is adaptable to the thermal environment of an externally mounted shuttle radiator.

(2) Fabrication of radiator modules is feasible with tape lay-up techniques where processing methods developed on this program are employed.

(3) Graphite/aluminum composites must await extensive scale-up development prior to application to space hardware application.

The relatively high conductivity (50-80 BTU/Hr. Ft. °F) as compared with other candidate materials such as titanium (4-5) enhances the performance - weight effectiveness of the radiator by reducing the number of fluid flow paths required. Excursions through 100 thermal excursions produced no material degradation and unidirectional strength through the full temperature range remained high, thus enhancing the structural - weight effectiveness.

The fact that readily available aerospace equipment and conventional process techniques can be applied to the fabrication of large panels will weigh heavily on future acceptance of the metal-matrix composite as a producible and reliable product. This work contributed to the concept of design and manufacturing flexibility as well as to composite materials technology.

Graphite/aluminum composites hold considerable promise for the future but the data generated on this program should be expanded to determine the parameters necessary to fabricate sound, consistent tape. Once the tape is satisfactory, the secondary fabrication will be straightforward. Figure 41 demonstrates that a high modulus graphite filaments can be diffusion bonded in an aluminum matrix. To reduce the process to practice appears promising. Aside from the potentially better conductivity in graphite/aluminum, the ability to perform machining operations with no more difficulty than conventional aluminum will afford a significant cost advantage. Furthermore, high modulus graphite fibers are likely to be much lower in cost than boron in the near future.

Recommended for future work are:

(1) Optimize the time-temperature-pressure parameters based on the process data evolved during this program relative to tape lay-ups for large panel constructions.

(2) Scale up to radiator panels of sufficient size to investigate the manufacturing problem areas; in particular, the tooling required to maintain uniformity of temperature over bond areas.

TABLE I COMPARISON OF CANDIDATE PANEL MATERIALS

Material	Density Pounds/cu in	Specific Strength 10^6	R.T. Specific Modulus 10^6	800°F Specific Strength 10^6	Availability	Manufacturing Characteristics	Thermal Conductivity BTU/HR-FT^2-FT-°F
1. Aluminum Alloy	(.1)	good (.5-.7)	100	.01	good	good	High 105
2. Titanium	(.16)	good (1.2)	110	.62	good	some problems	Low 4-6
3. Stainless Steel	(.28)	good (.6)	100	.2-.3	good	good	Low 8-12
4. Aluminum Composite 35 V/O Boron	(.094)	good (1.0-1.4)	200	1.0-1.4	pilot prod.	developmental	High 50-80*
5. Aluminum Composite 25 V/O Graphite	(.095)	over 1.5 Expected	over 300 Expected	over 1.5 Expected	early developmental	probably good	Probably high

*The first thermal conductivity data is reported herein

TABLE II

EFFECT OF TEST TEMPERATURE ON TENSILE PROPERTIES
OF BORON-ALUMINUM COMPOSITES, 7 PLY UNDIRECTIONAL (30-35 V/O BORON)
AND 6 PLY BORON + 2 PLY STAINLESS STEEL

PANEL NO.	TEST TEMPERATURE	Ftu Ksi	TEST DIRECTION
26 B	R. T.	79.8	Parallel to
26 B	R. T.	75.9	boron
26 B	400 °F	95.0	Parallel to
26 B	400 °F	81.4	boron
26 B	800 °F	70.8	Parallel to
26 B	800 °F	84.9	boron
28	-320 °F	72.1	Parallel to
28	-320 °F	70.2	boron
26B	R. T.	13.7	Transverse
26B	R. T.	14.0	to boron
26A	R. T.	14.3	
26B	400 °F	11.3	Transverse
26B	400 °F	10.9	to boron
26B	800 °F	4.3	Transverse
26B	800 °F	4.6	to boron
26A	-250 °F	15.3	Transverse
26A	-320 °F	14.8	to boron
	6 Ply + Two 90° Cross Plys St.St.		
25	R. T.	38.3	Parallel
25	400 °F	22.7	to boron

TABLE III

COMPRESSIVE STRENGTH OF COMPOSITES, 7 PLY BORON-ALUMINUM (30-35 V/O FILAMENT) AND 6 PLY BORON + 2 PLY STAINLESS STEEL

PANEL NO.	TYPE COMPOSITE	TEST DIRECTION	COMPRESSION LOAD AT FAILURE Ksi
29	B/Al	Parallel to boron	184.0
29	B/Al		169.0
29	B/Al		160.0
27B	B/Al	Transverse to	20.0
27B	B/Al	boron	21.9
27B	B/Al		21.5
25	B/Al/StSt.	Parallel to boron	83.4
25	B/Al/StSt.		88.3

TABLE IV

EFFECT OF TEST TEMPERATURE ON FLEXURAL STRENGTH OF 7 PLY UNIDIRECTIONAL BORON/ALUMINUM COMPOSITES (30-35 V/O)

PANEL NO.	TEST TEMPERATURE °F	TEST DIRECTION *	Fb Ksi
28	R. T.	Transverse	28.4
28	R. T.	Transverse	27.0
23	R. T.	Parallel	74.7
23	R. T.	Parallel	65.2
28	400	Transverse	25.1
28	400	Transverse	25.9
23	400	Parallel	106.3
23	400	Parallel	115.8
28	800	Transverse	9.3
28	800	Transverse	10.5
23	800	Parallel	106.1
29	800	Parallel	114.3
28	-250	Transverse	18.6
28	-250	Transverse	21.0
23	-250	Parallel	94.2
23	-250	Parallel	88.1

* Relative to boron filament direction.

TABLE V

EFFECT OF TEST TEMPERATURE ON FLEXURAL STRENGTH OF COMPOSITES, 6 PLY BORON + 2 MIDDLE 90° CROSS PLIES STAINLESS STEEL

PANEL NO.	TEST TEMPERATURE °F	TEST DIRECTION *	Fb Ksi
25	R. T.	Transverse	30.5
25	R. T.	Transverse	27.6
24	R. T.	Parallel	63.9
24	R. T.	Parallel	73.5
25	400	Parallel	79.9
25	400	Parallel	62.2
25	800	Parallel	84.6
25	800	Parallel	89.6
25	-250	Parallel	84.3
25	-250	Parallel	82.8

* Relative to boron filament direction.

TABLE VI

EFFECT OF THERMAL CYCLING ON FLEXURAL PROPERTIES
OF 7 PLY UNDIRECTIONAL BORON/ALUMINUM COMPOSITES (30-35 V/O BORON)
(PANEL 23) AND 6 PLY BORON + 2 CROSS PLY STAINLESS STEEL (PANEL 24)

PANEL NO.	THERMAL EXPOSURE	Fb* Ksi	E $X10^6$ PSI
23		74.7	28.5
23	None	65.2	30.1
24		63.9	17.9
24		73.5	18.1
23	-250 to 800°F	84.6	26.6
23	100 times	120.0	27.3
24		73.3	19.9
24		87.0	17.7

* Tested parallel to boron filament

TABLE VII

EFFECT OF TEST TEMPERATURE ON FLEXURAL MODULUS OF 7 PLY UNIDIRECTIONAL BORON/ALUMINUM COMPOSITES (30-35 V/O) AND 6 PLY + 2 CROSS PLY STAINLESS STEEL

7 PLY BORON/ALUMINUM

PANEL NO.	TEST TEMPERATURE °F	TEST DIRECTION TO BORON FILAMENTS	E PSI X 10^6
28	-250	Transverse	3.5*
28	-250	Transverse	1.9*
23	-250	Parallel	23.4
23	-250	Parallel	26.3
28	R.T.	Transverse	3.7*
28	R.T.	Transverse	4.3*
23	R.T.	Parallel	28.5
23	R.T.	Parallel	30.1
28	400	Transverse	1.5*
28	400	Transverse	0.8*
23	400	Parallel	22.4
23	400	Parallel	22.7
28	800	Transverse	0.4*
28	800	Transverse	0.5*
23	800	Parallel	13.1*
29	800	Parallel	13.3*

6 PLY + 2 CROSS PLY STAINLESS STEEL

25	-250	Parallel	18.0
25	-250	Parallel	19.8
24	R.T.	Parallel	17.8
24	R.T.	Parallel	17.9
25	R.T.	Transverse	2.3*
25	R.T.	Transverse	2.0*
25	400	Parallel	16.2
25	400	Parallel	10.2
25	800	Parallel	7.1*
25	800	Parallel	6.4*
Titanium	R.T.		16.0
	400		14.0

* Specimens showed shearing deformation during testing. Calculated modulus values are included for comparison purposes only.

TABLE VIII

PROCESS FOR NICKEL COATING OF GRAPHITE FILAMENTS

1.	Heat Cleaning*	Vacuum diffusion furnace run at 1500°F, 1 hour, inert atmosphere.
2.	Sensitizing Treatment	Immersion in mixture of Stannous Chloride and Hydrochloric acid for 1 minute.
3.	Activation Treatment	Immersion in mixture of Palladium Chloride and Hydrochloric acid for 1 minute.
4.	Nickel Coating	Immersion in mixture of standard electroless nickel solution at 120° to 180°F (McDermid proprietary solution).

The process required careful handling of the filaments so as not to blanket areas with holding devices but continuous or periodic rotation over a mandrel will permit all over coating. Unlike electroplating processes, relatively thick bundles of filaments can be coated throughout. All solutions should be allowed to wet all filament surfaces, however.

* Not required unless epoxy or similar coatings have been used to size filaments and which have not been removed by the supplier of filaments.

TABLE IX

THERMAL CONDUCTIVITY AT ROOM TEMPERATURE

Specimen	Figure	K_U	K_L	K_C(Measured)
1	34	64	41.8	46
2	36	76.8	39.9	56
3	35	76.8	76.8	73
4	37	73.8	67.7	74

Note that for specimen 3 $t_x = 0$, $\frac{V_x}{V_T} = 0$, so the equation for K_L reduces to equal the equation for K_U.

NOTE: Thermal expansion was not measured.
It has been reported (reference 8)
Parallel: 3.2 in/in °F x 10^6
Perpendicular: 10 in/in °F x 10^6

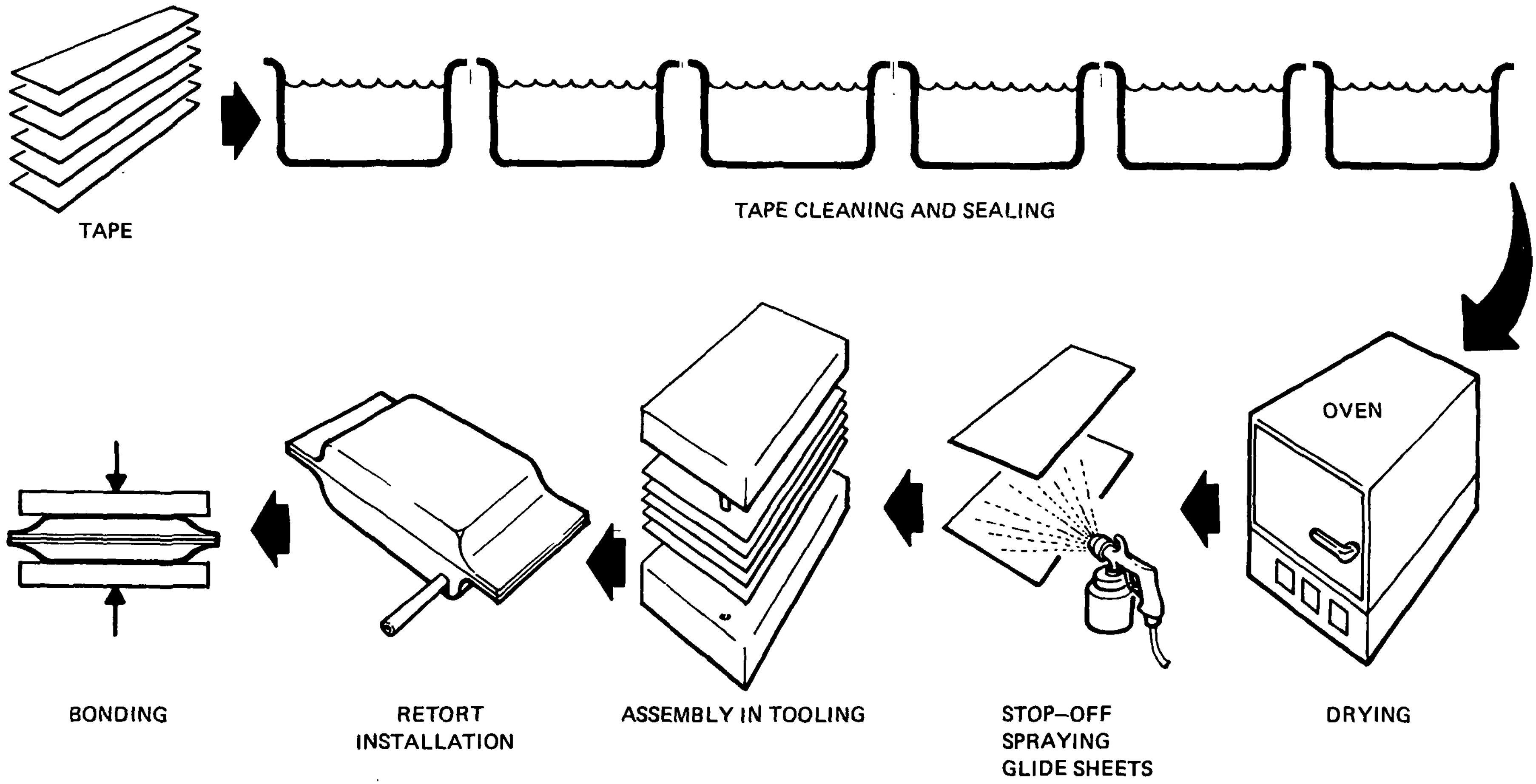
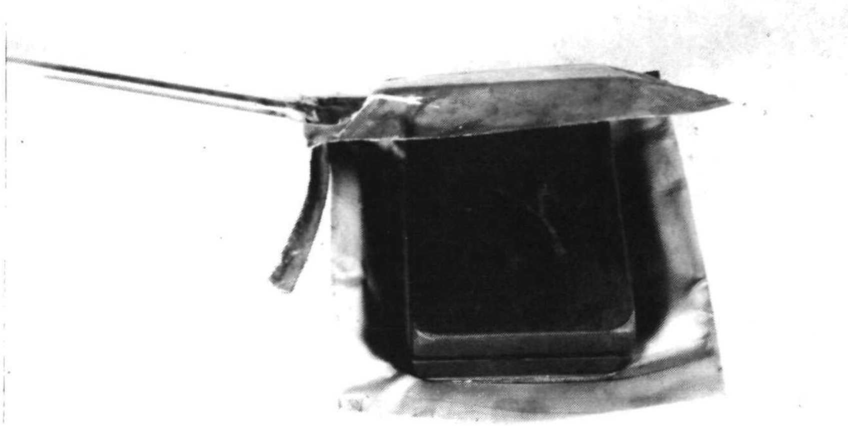


FIGURE 1   FLOW SHEET OF SECONDARY/DIFFUSION BONDING PROCESS



OPENED RETORT, PRESSURE PLATES IN POSITION

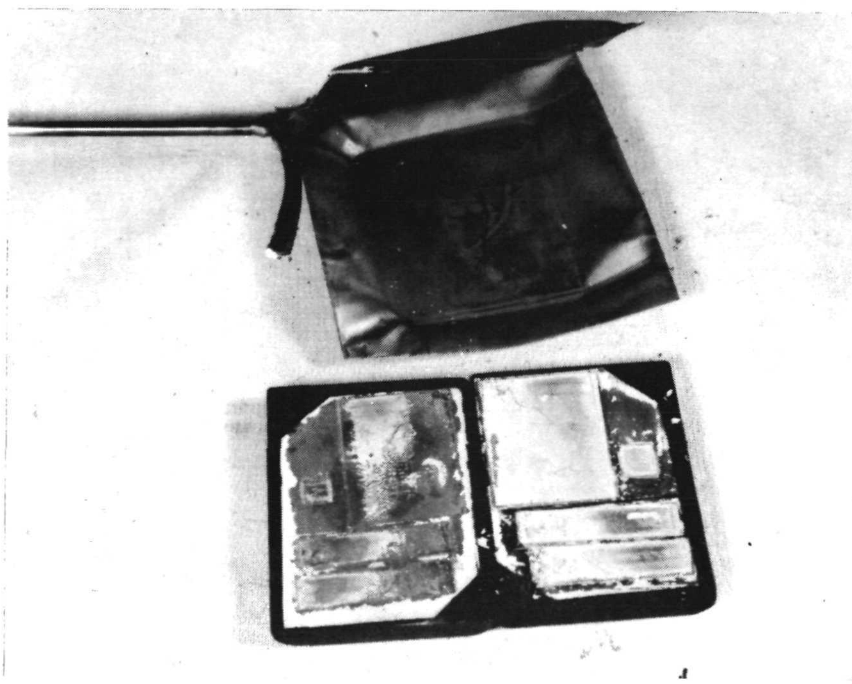


FIGURE 2 PRESSURE PLATES OPENED TO EXPOSE BONDED SPECIMENS (ON RIGHT) AND GLIDE SHEETS

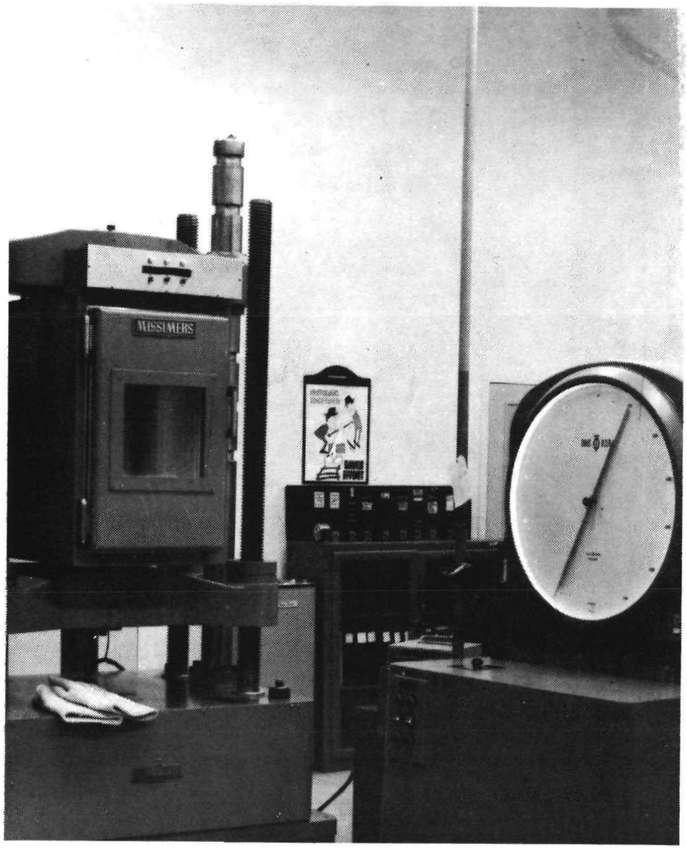


FIGURE 3 ELEVATED TEMPERATURE TEST APPARATUS

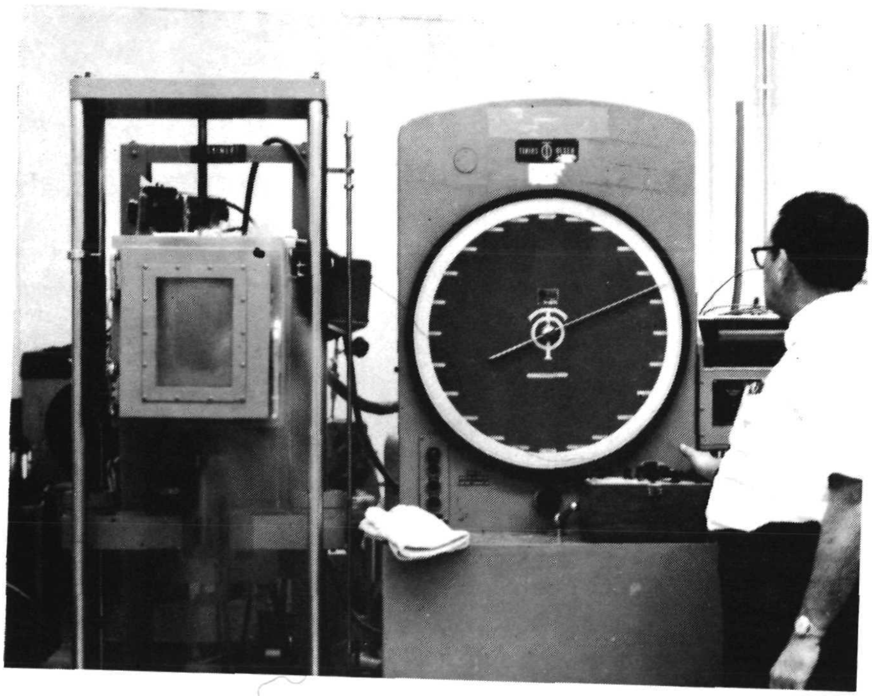


FIGURE 4 TENSILE TESTING AT –250 °F IN COLD BOX APPARATUS

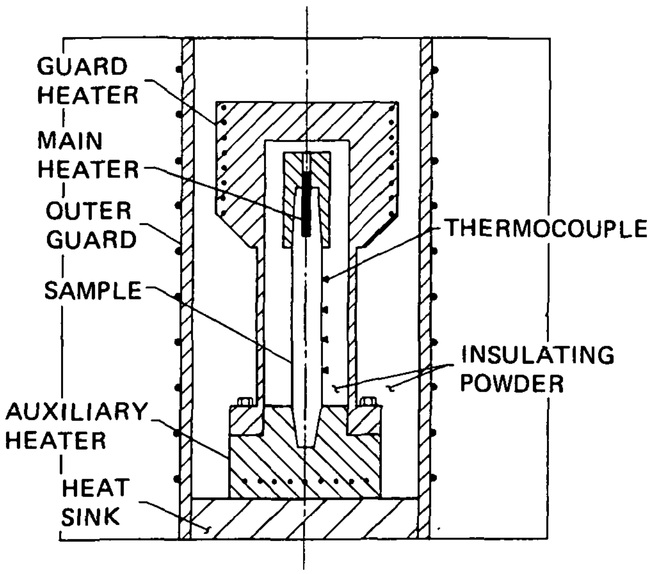


FIGURE 5 SCHEMATIC OF DYNATECH AXIAL ROD THERMAL CONDUCTIVITY INSTRUMENT MODEL TCAGM SERIES

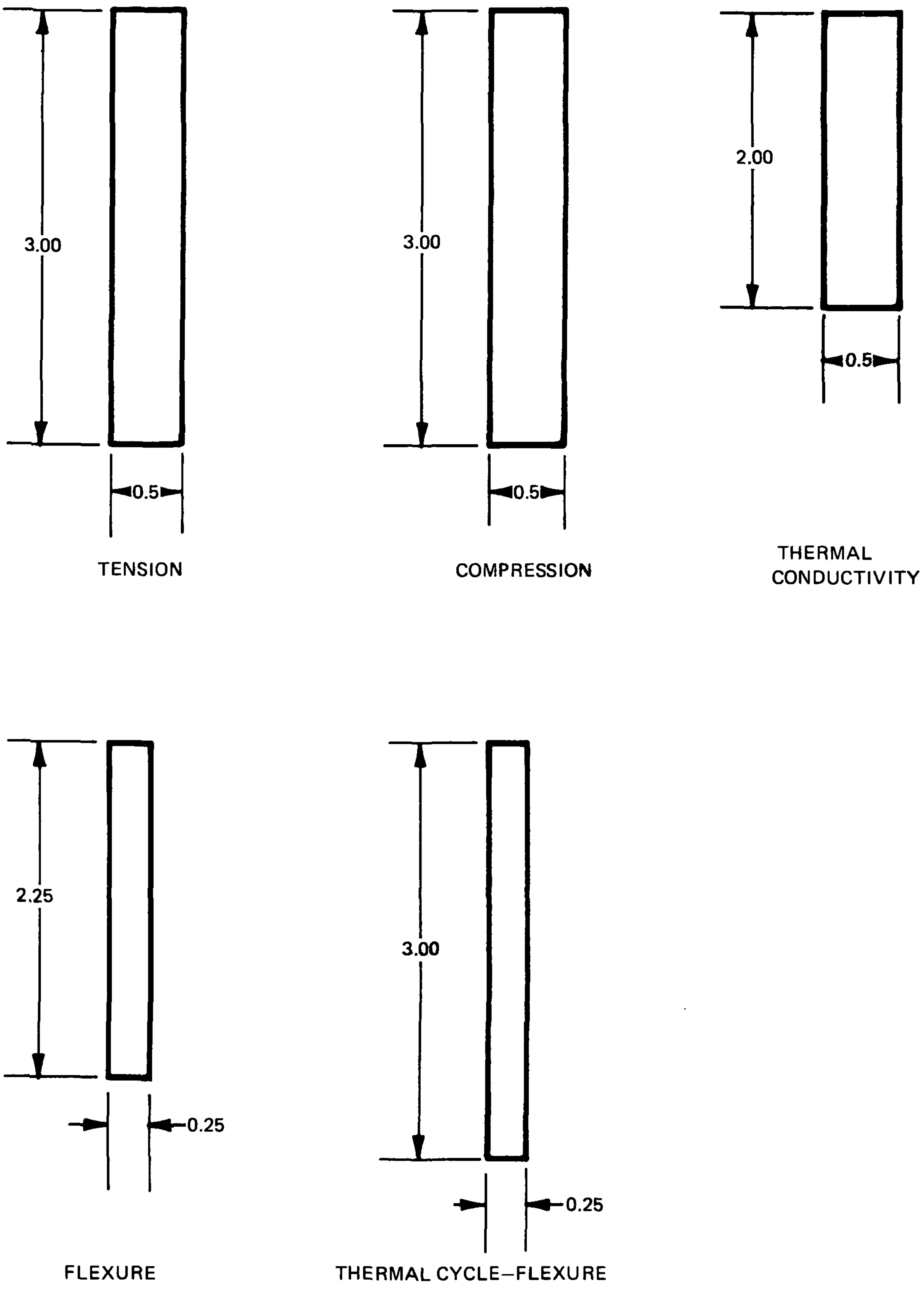


FIGURE 6 MATERIAL PROPERTY TEST SPECIMEN CONFIGURATIONS

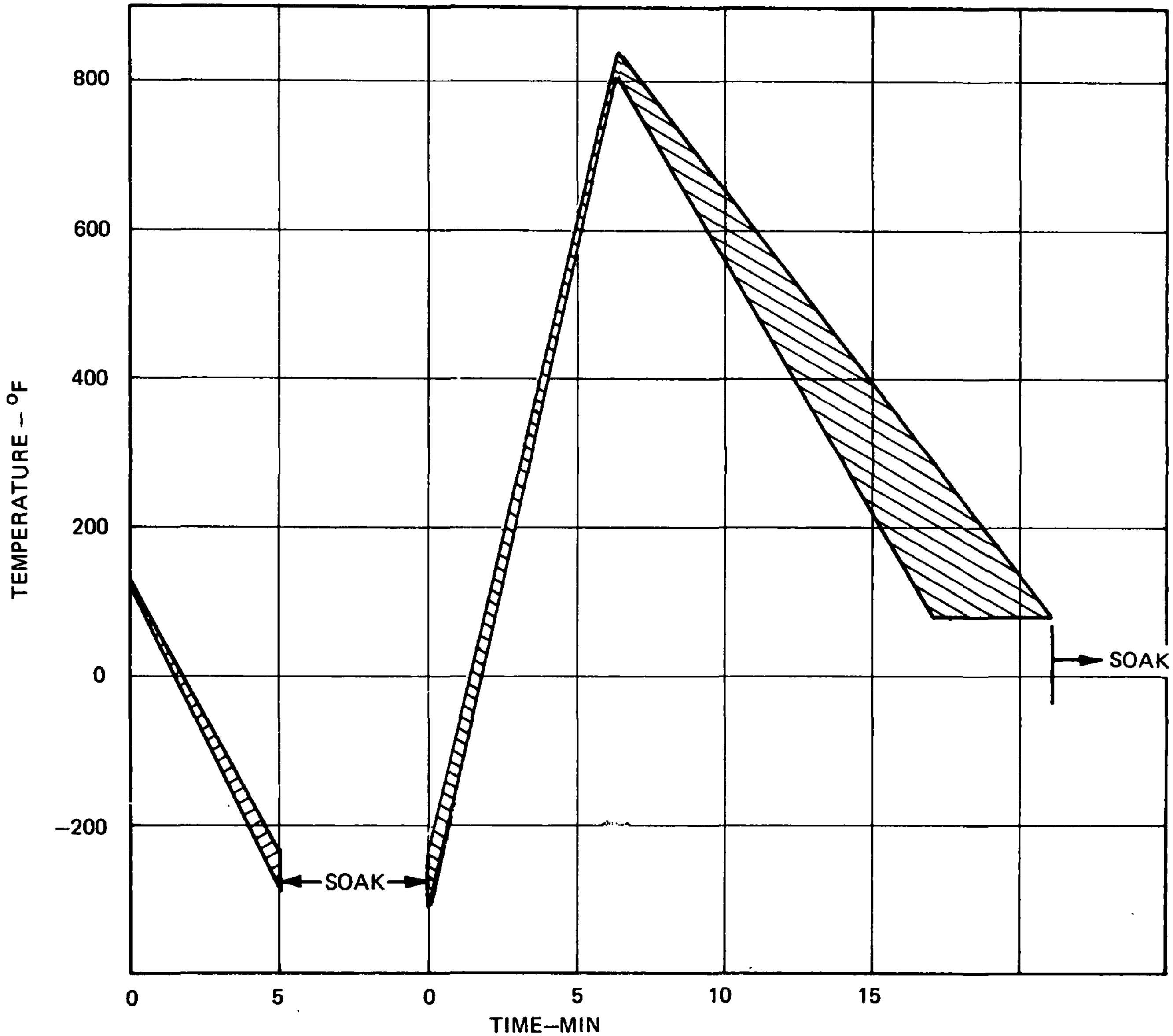


FIGURE 7 THERMAL CYCLING SCHEDULE, EXTERNALLY MOUNTED RADIATOR, TYPICAL OF LIMIT CASE OF A REENTRY FROM ORBIT

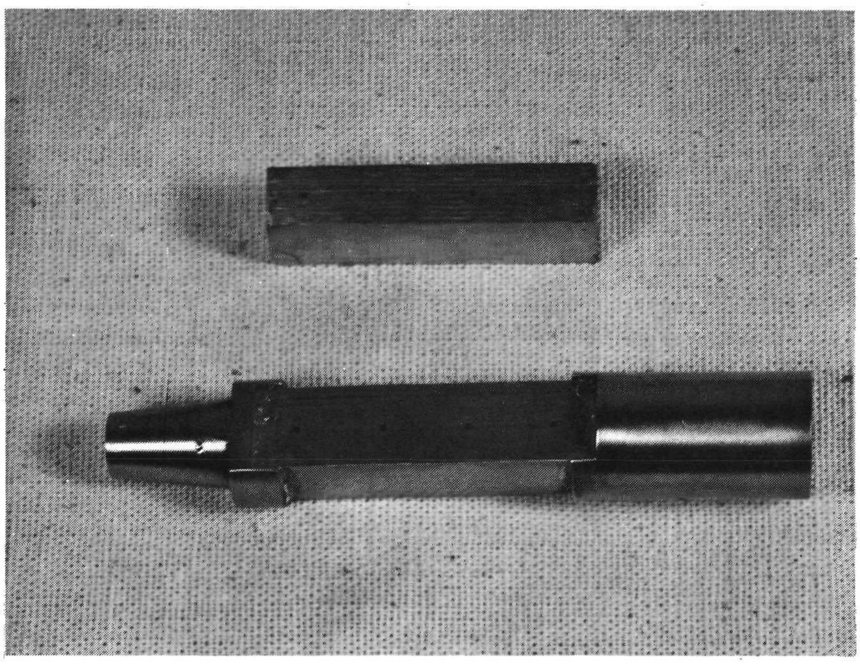


FIGURE 8 THERMAL CONDUCTIVITY SPECIMEN

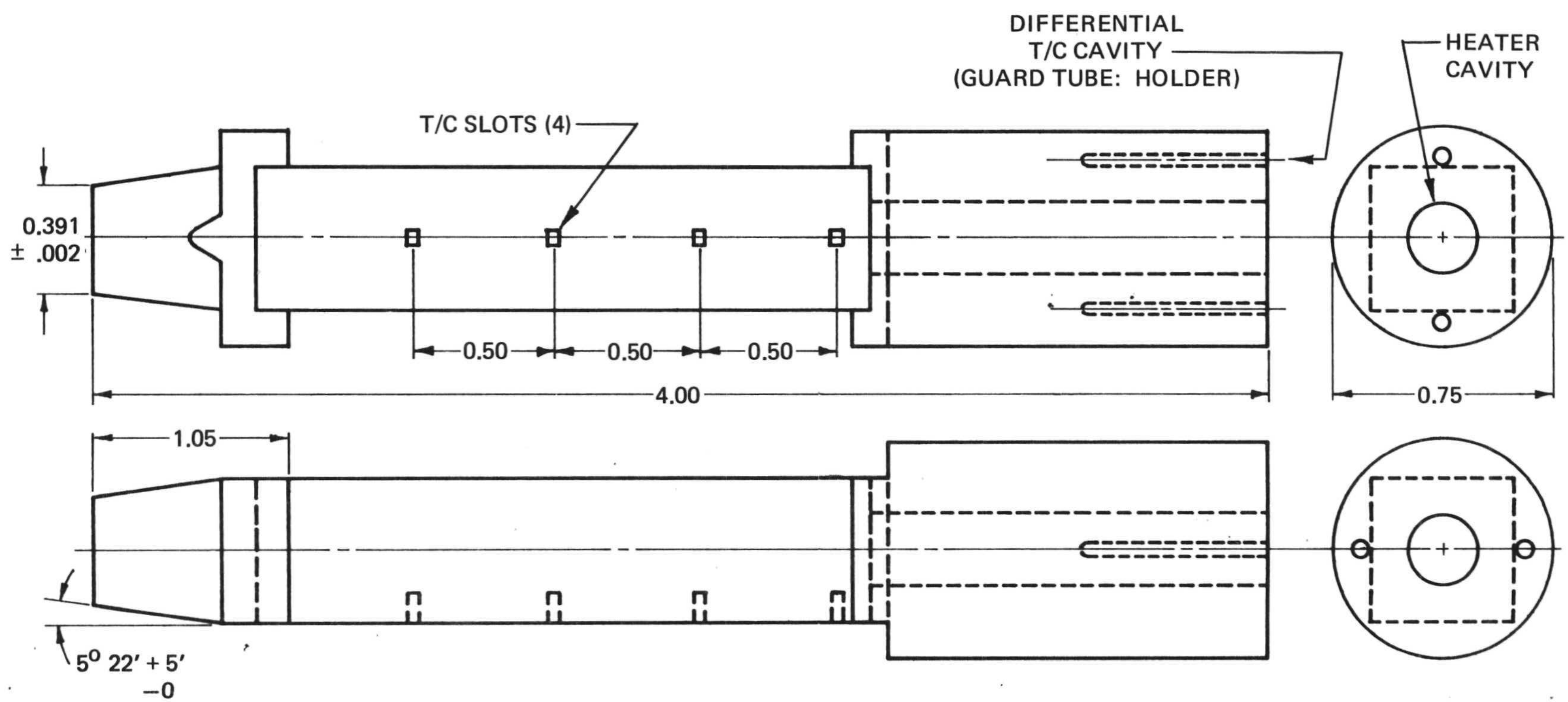


FIGURE 9 THERMAL CONDUCTIVITY TEST SPECIMEN CONFIGURATION

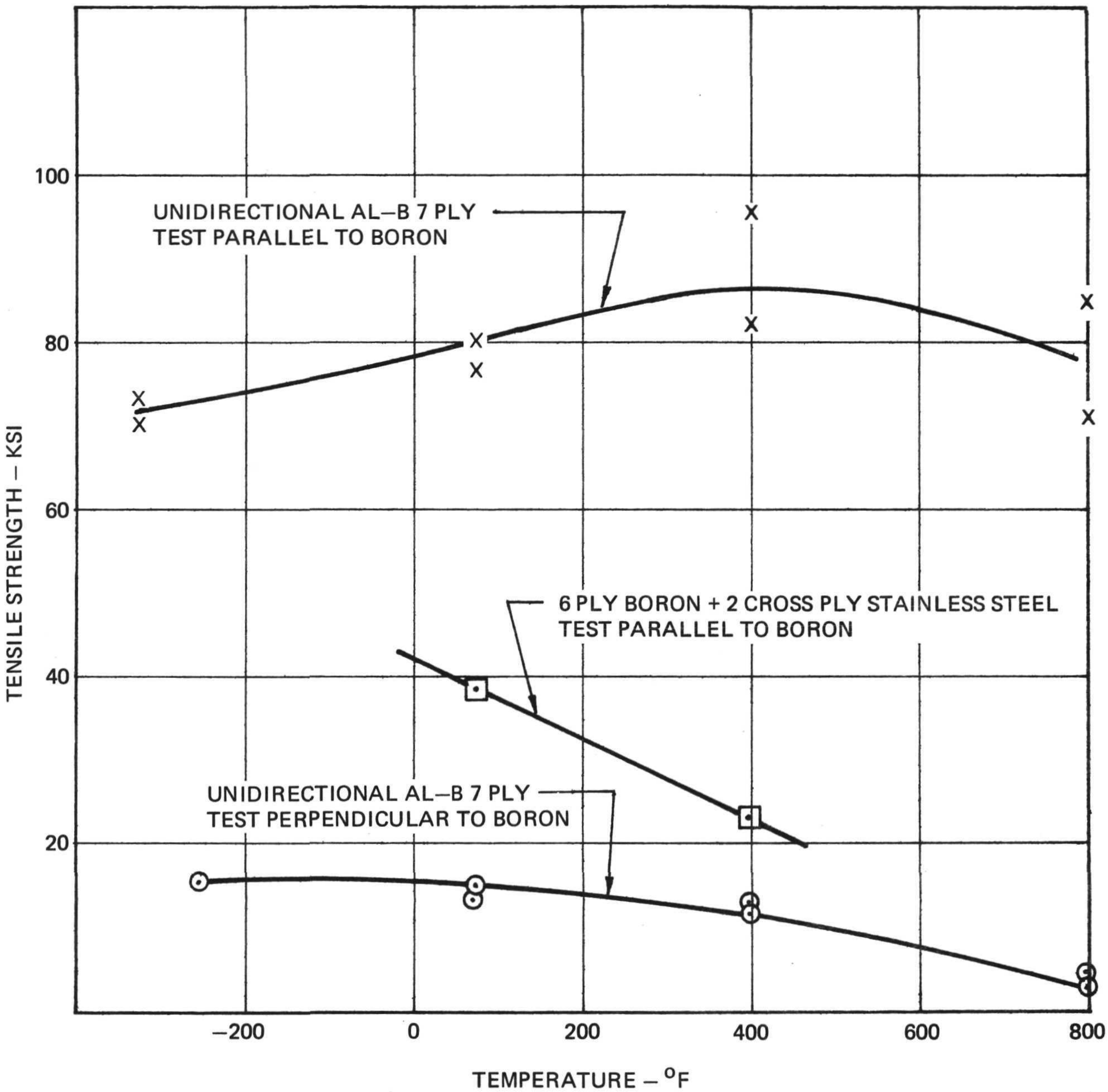


FIGURE 10 TENSILE STRENGTH VARIATION WITH TEMPERATURE – 7 PLY UNIDIRECTIONAL BORON/ALUMINUM (30 – 35 V/O B)

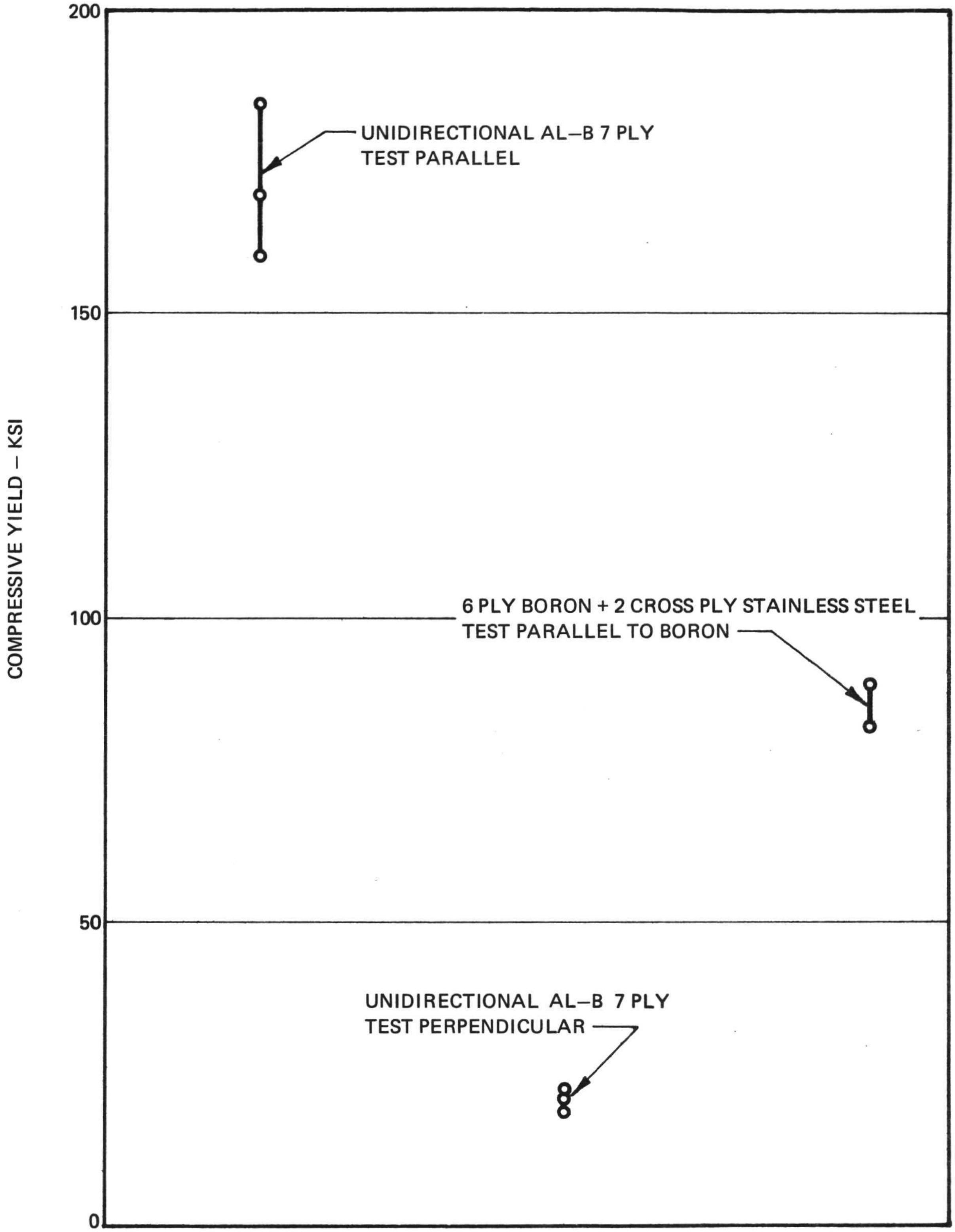


FIGURE 11 COMPRESSIVE STRENGTH VARIATION WITH COMPOSITE FORM AND DIRECTION OF FILAMENTS

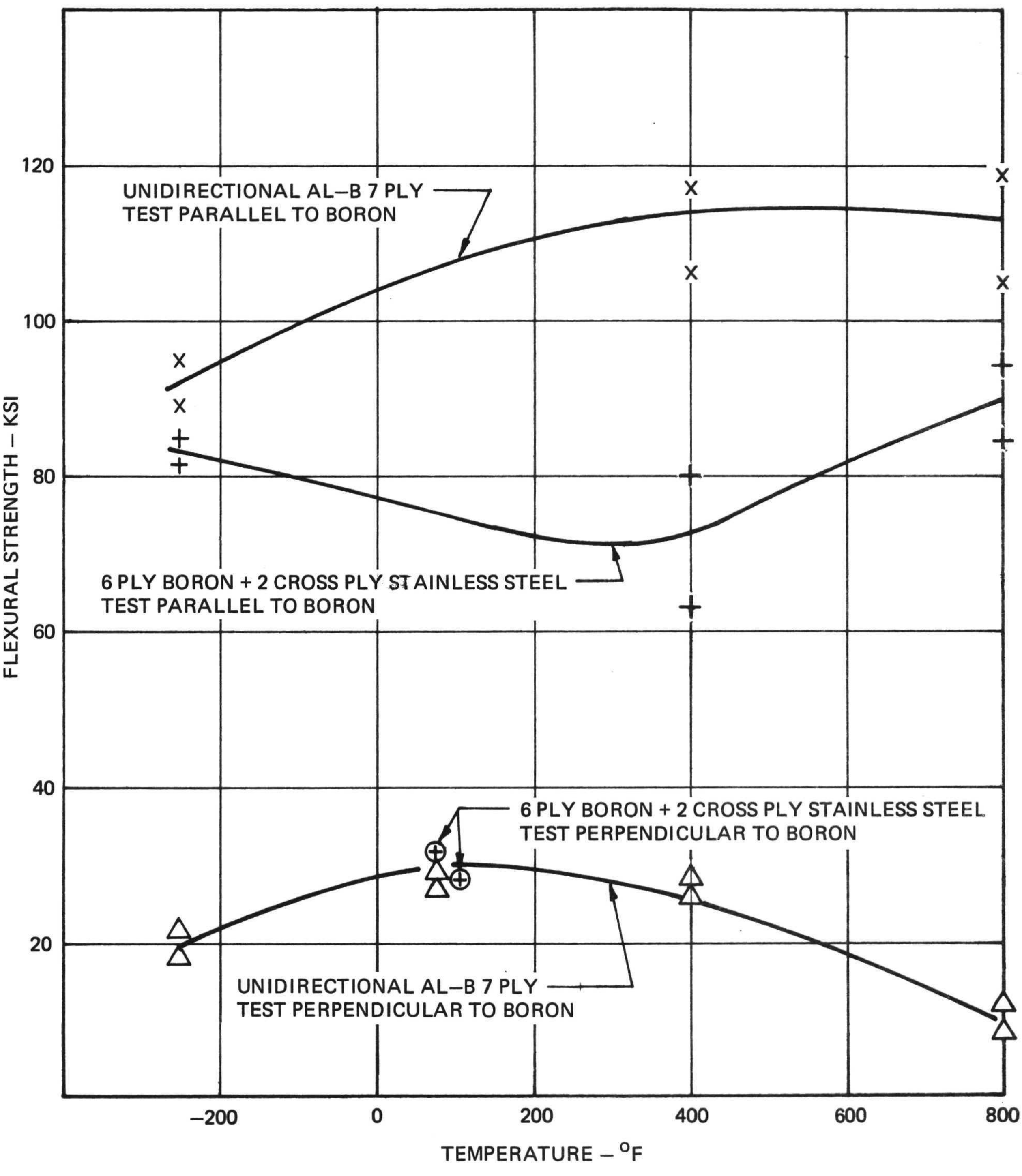


FIGURE 12 FLEXURAL STRENGTH VARIATION WITH TEMPERATURE

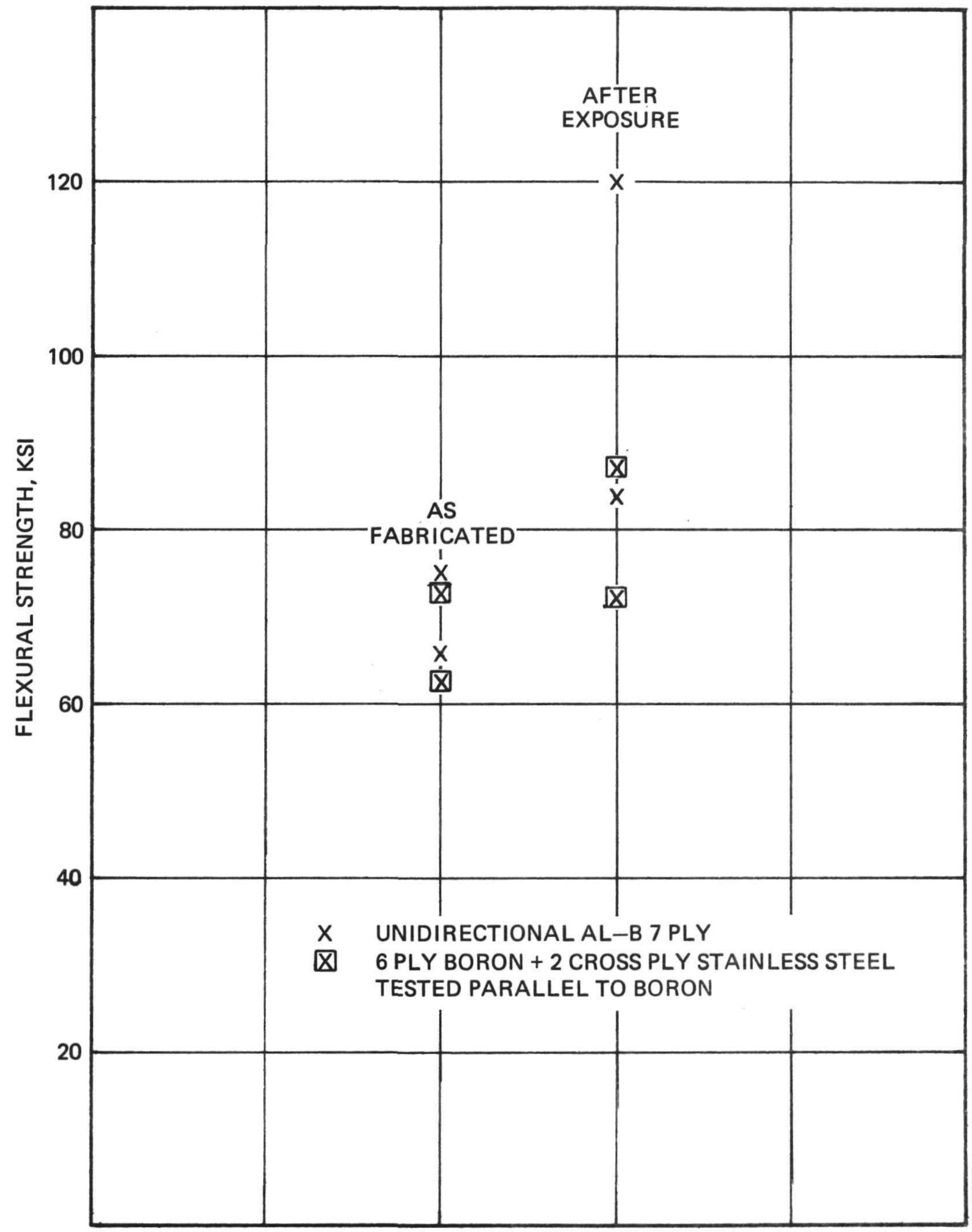


FIGURE 13 ROOM TEMPERATURE FLEXURAL STRENGTH, EFFECTS OF 100 CYCLES EXPOSURE BETWEEN –250° TO 800°F

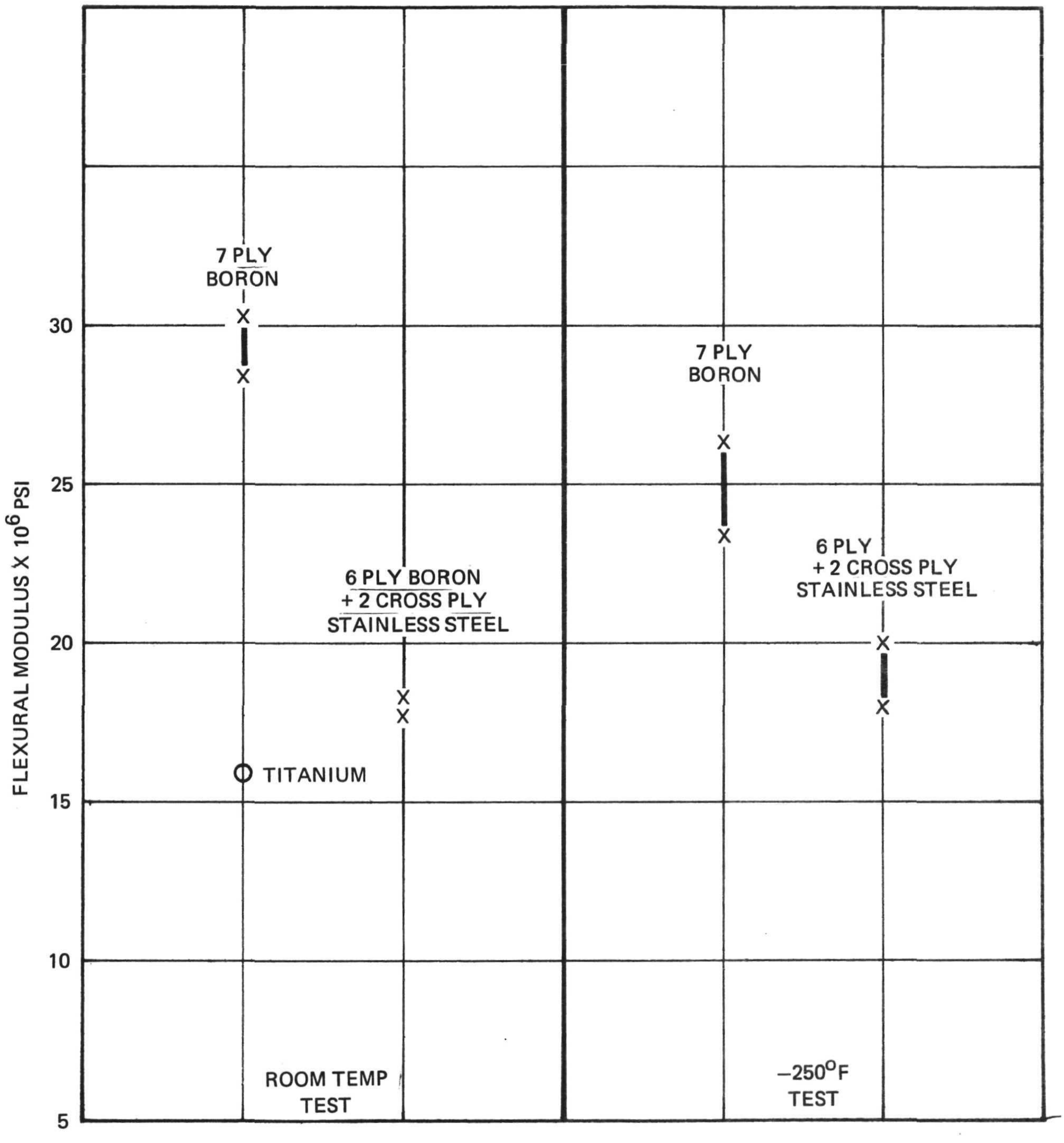


FIGURE 14 FLEXURAL MODULUS VARIATION WITH COMPOSITE LAYUP, R.T. AND −250°F TESTED PARALLEL TO BORON

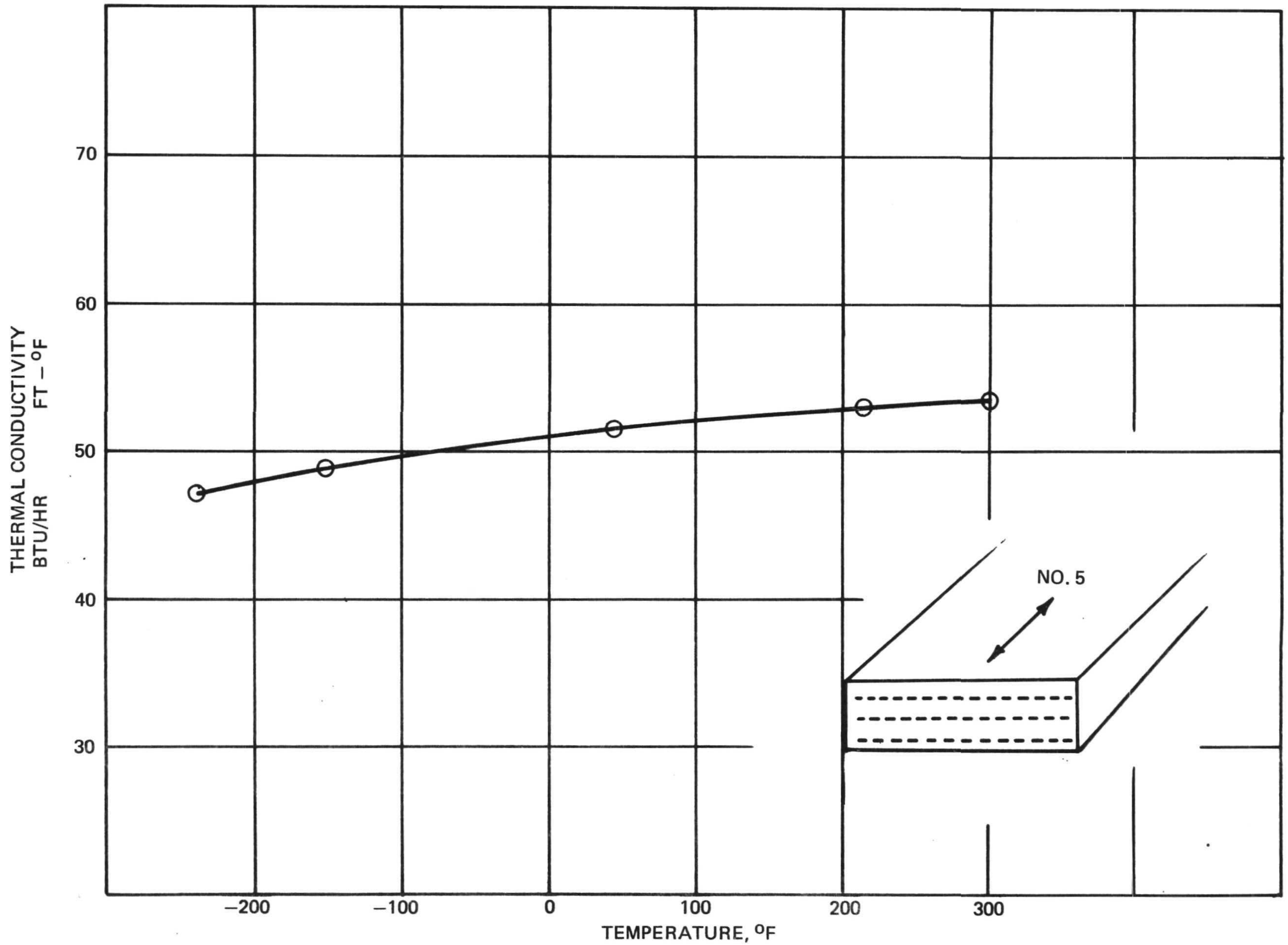


FIGURE 15 THERMAL CONDUCTIVITY OF GRAPHITE-ALUMINUM COMPOSITE (IN PLANE OF SHEET, 3 PARALLEL PLYS OF NICKEL COATED GRAPHITE TOW)

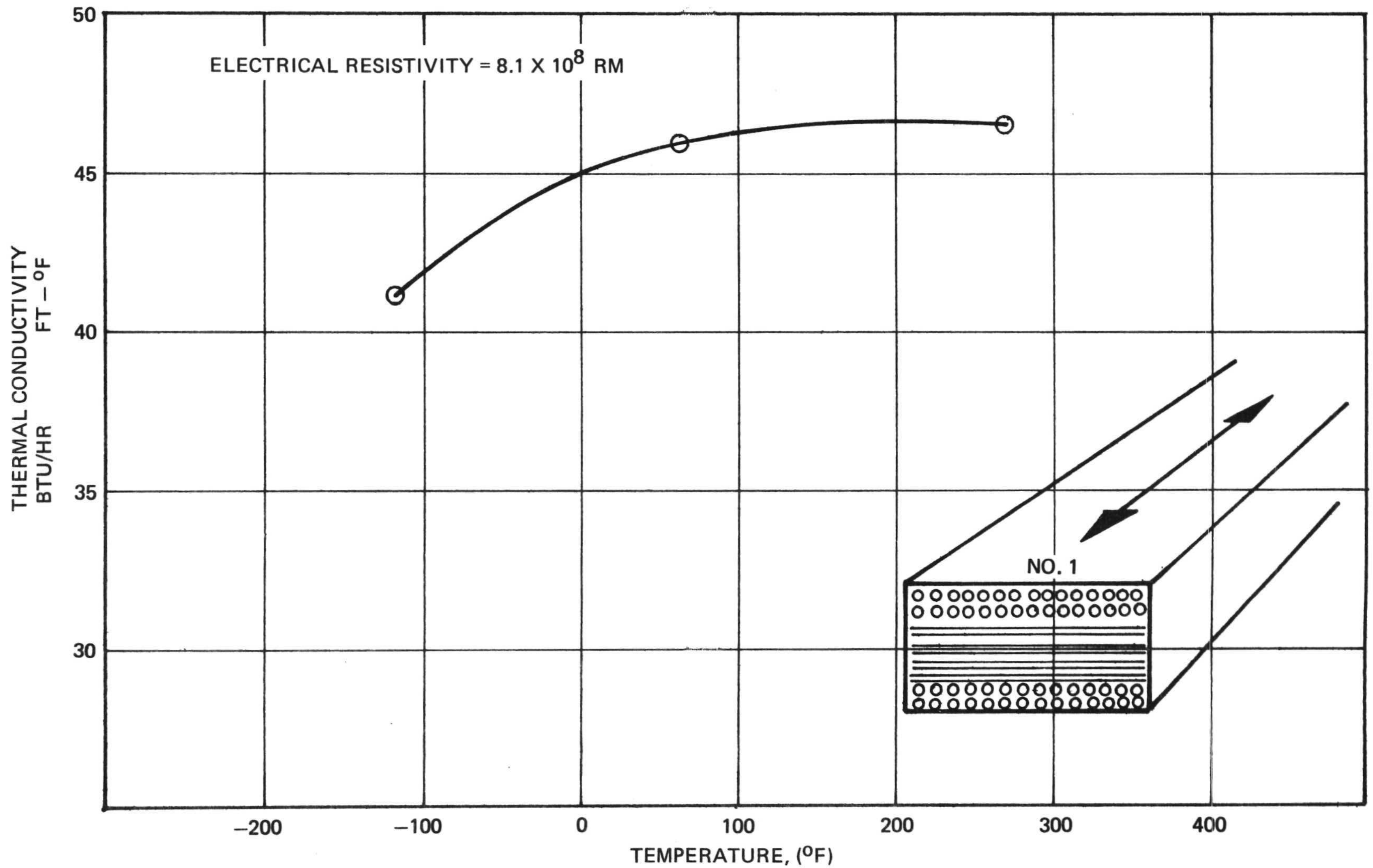


FIGURE 16 THERMAL CONDUCTIVITY OF BORON/ALUMINUM COMPOSITE (IN PLANE OF SHEET, 2 PARALLEL OUTER PLYS, FOUR 90° CENTER CROSS PLYS) (45 – 50 V/O B)

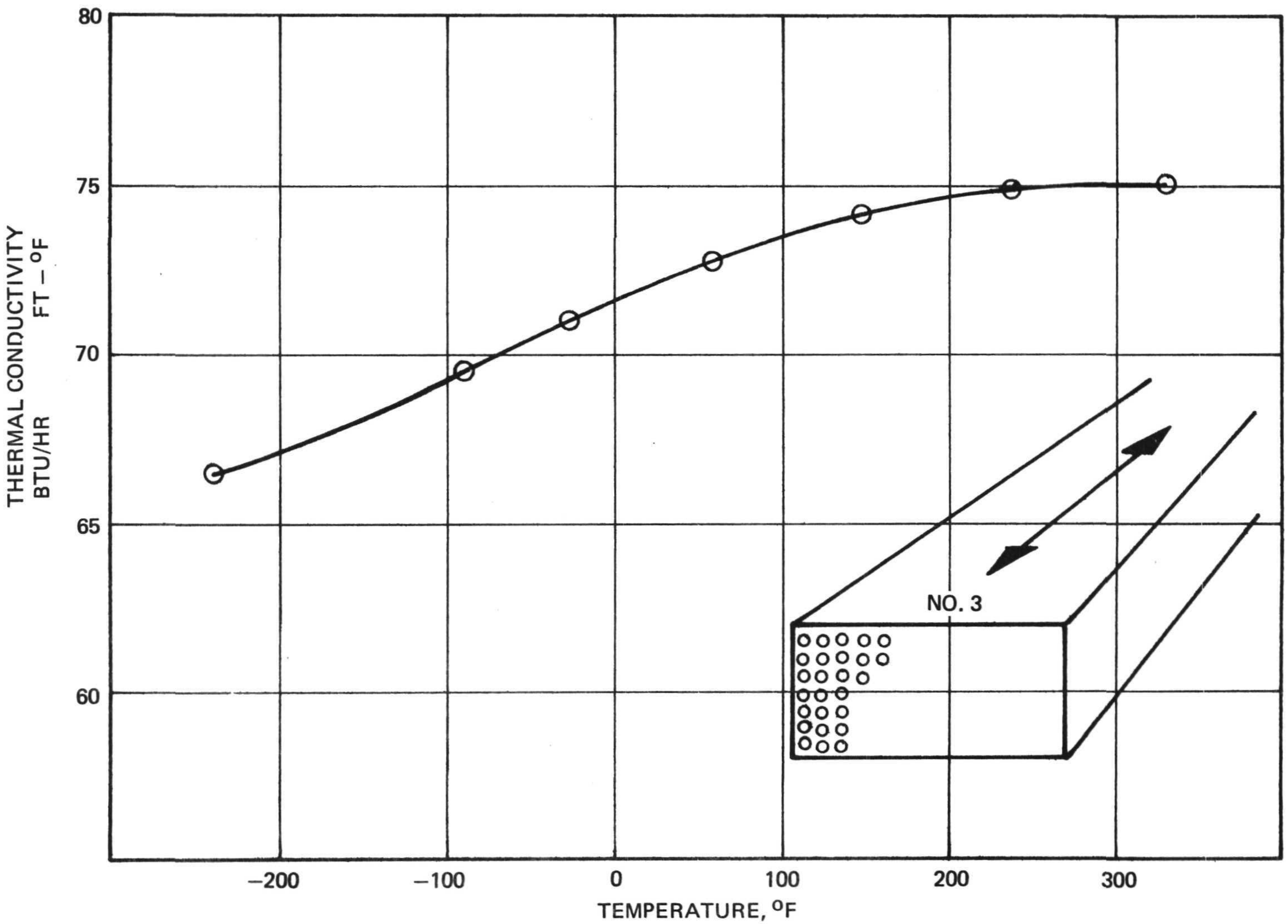


FIGURE 17 THERMAL CONDUCTIVITY OF BORON/ALUMINUM 7 PLY UNIDIRECTIONAL (30 – 35 V/O B)

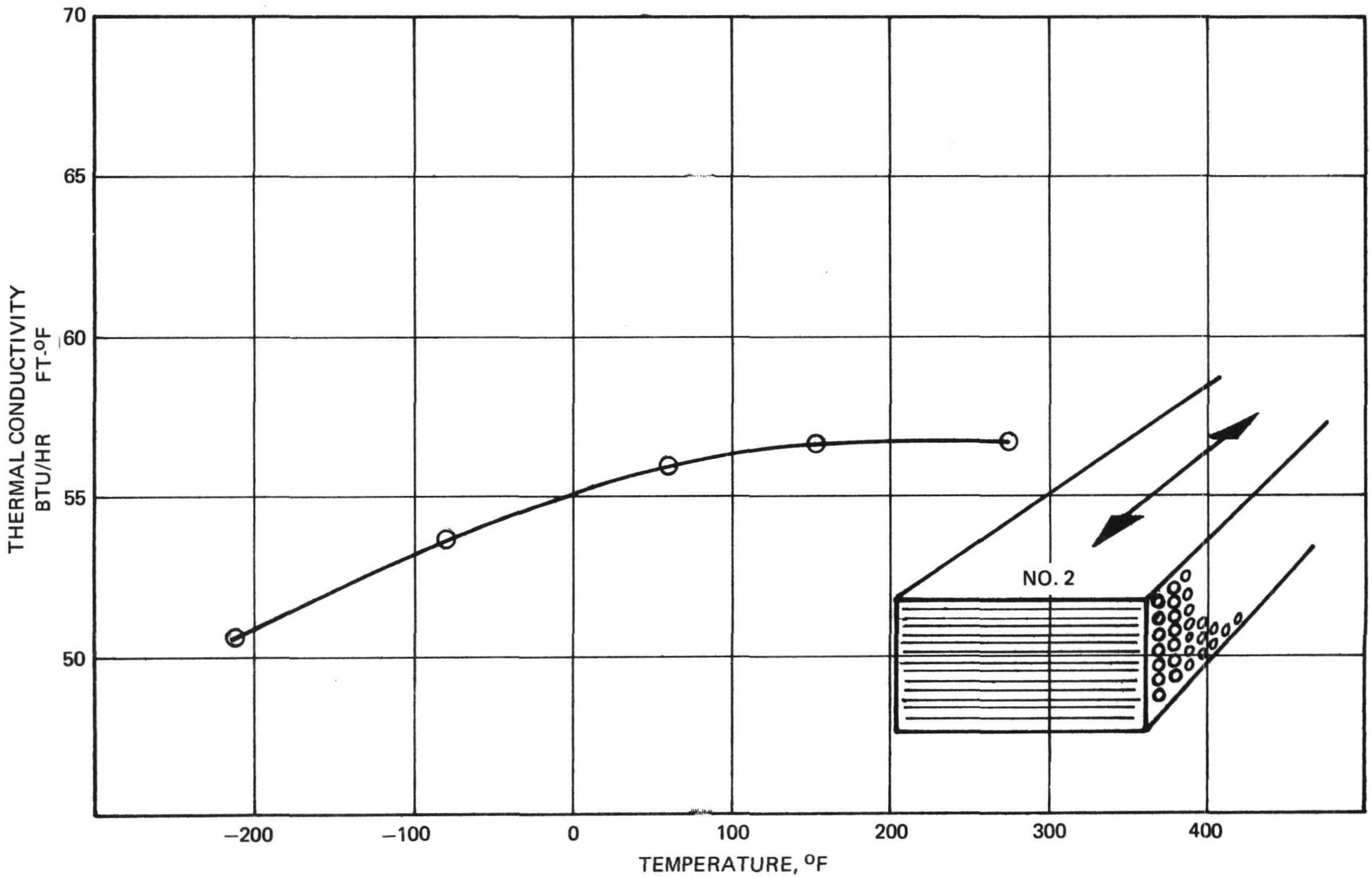


FIGURE 18 THERMAL CONDUCTIVITY TRANSVERSE AND IN PLANE OF 7 PLY UNIDIRECTIONAL B/AL (30 – 35 V/O B)

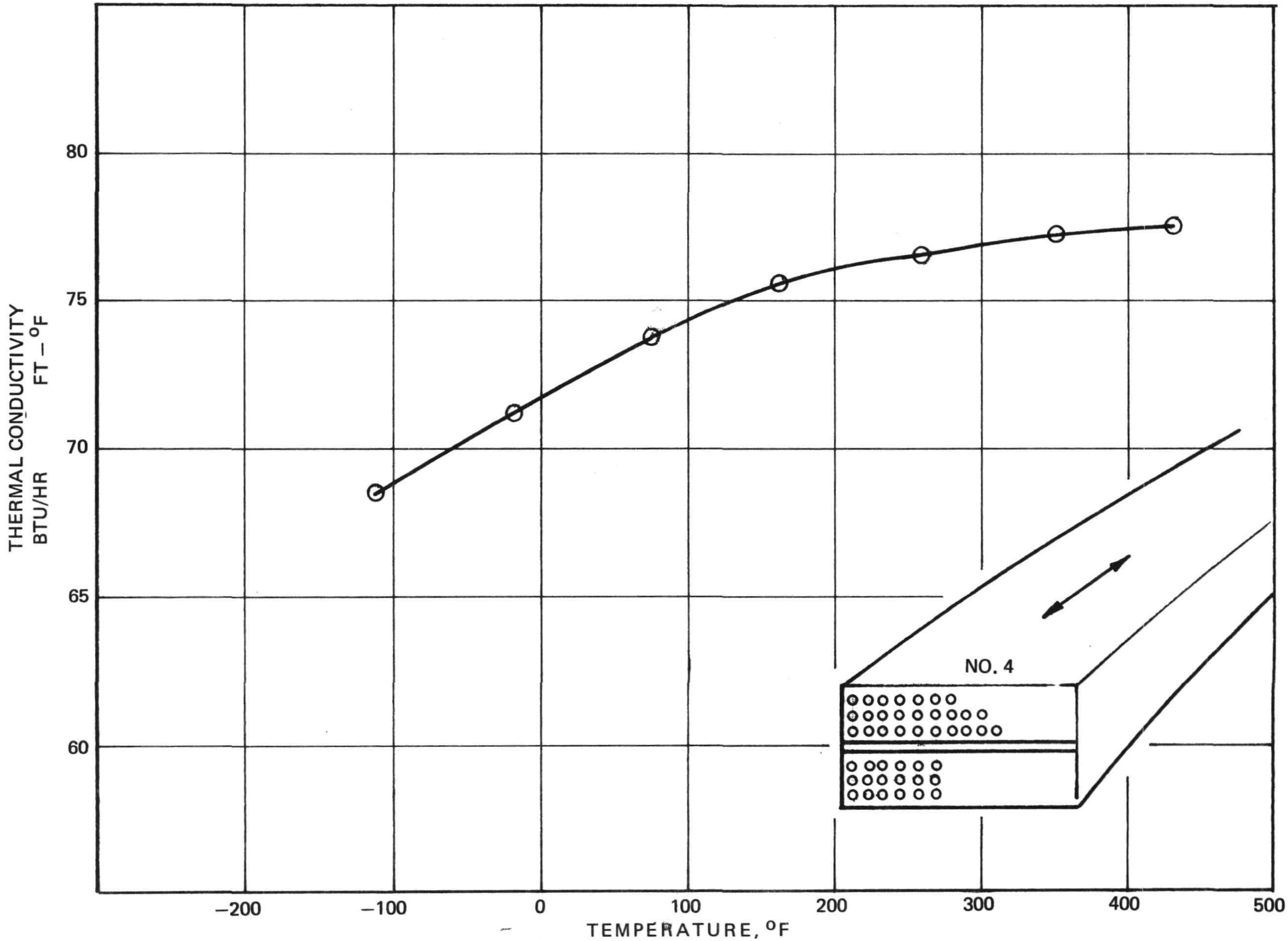


FIGURE 19 THERMAL CONDUCTIVITY OF BORON/ALUMINUM + STAINLESS STEEL CROSS PLY. (IN PLANE OF SHEET, 3 PARALLEL OUTER PLYS BORON 3 CENTER 90° CROSS PLYS STAINLESS STEEL) (30 – 35 V/O B)

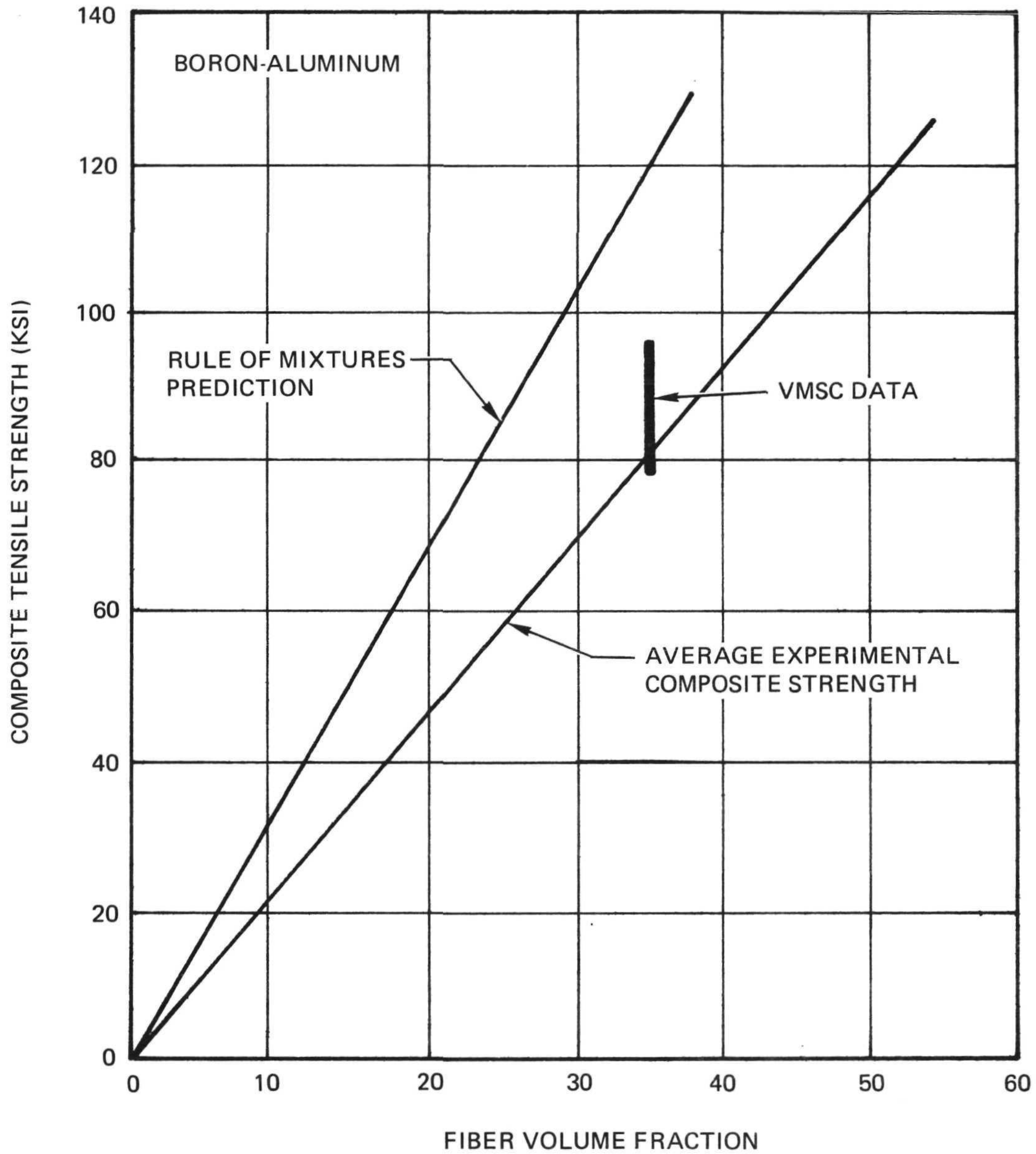
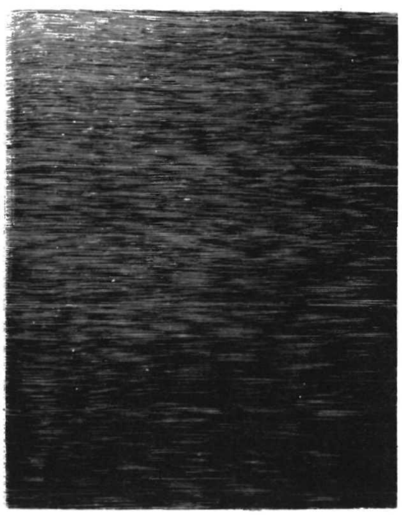


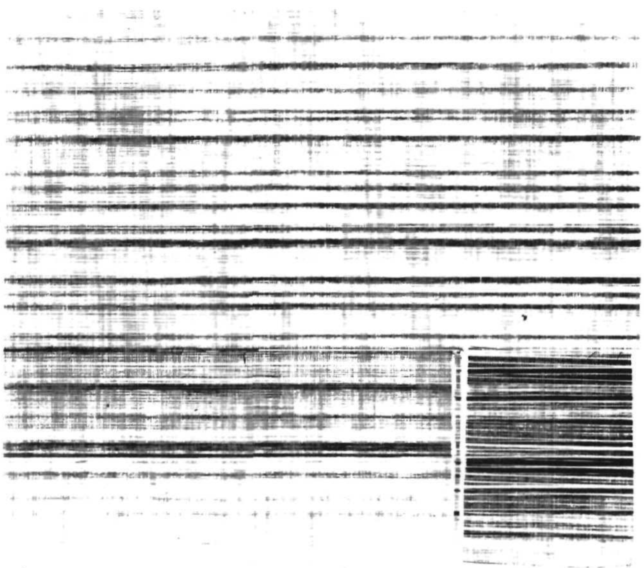
FIGURE 20 VARIATION OF COMPOSITE STRENGTH WITH VOLUME FRACTION (REF 5)

DB18



DB19

FIGURE 21 RADIOGRAPHS OF 7 PLY BORON/ALUMINUM PANELS (DB 19 WAS SPLICED PARALLEL TO FILAMENTS IN HALF INCH WIDE STRIPS)



DB25

FIGURE 22 RADIOGRAPH OF 7 PLY BORON/ALUMINUM PANEL WITH SPLICED CORNER PIECE

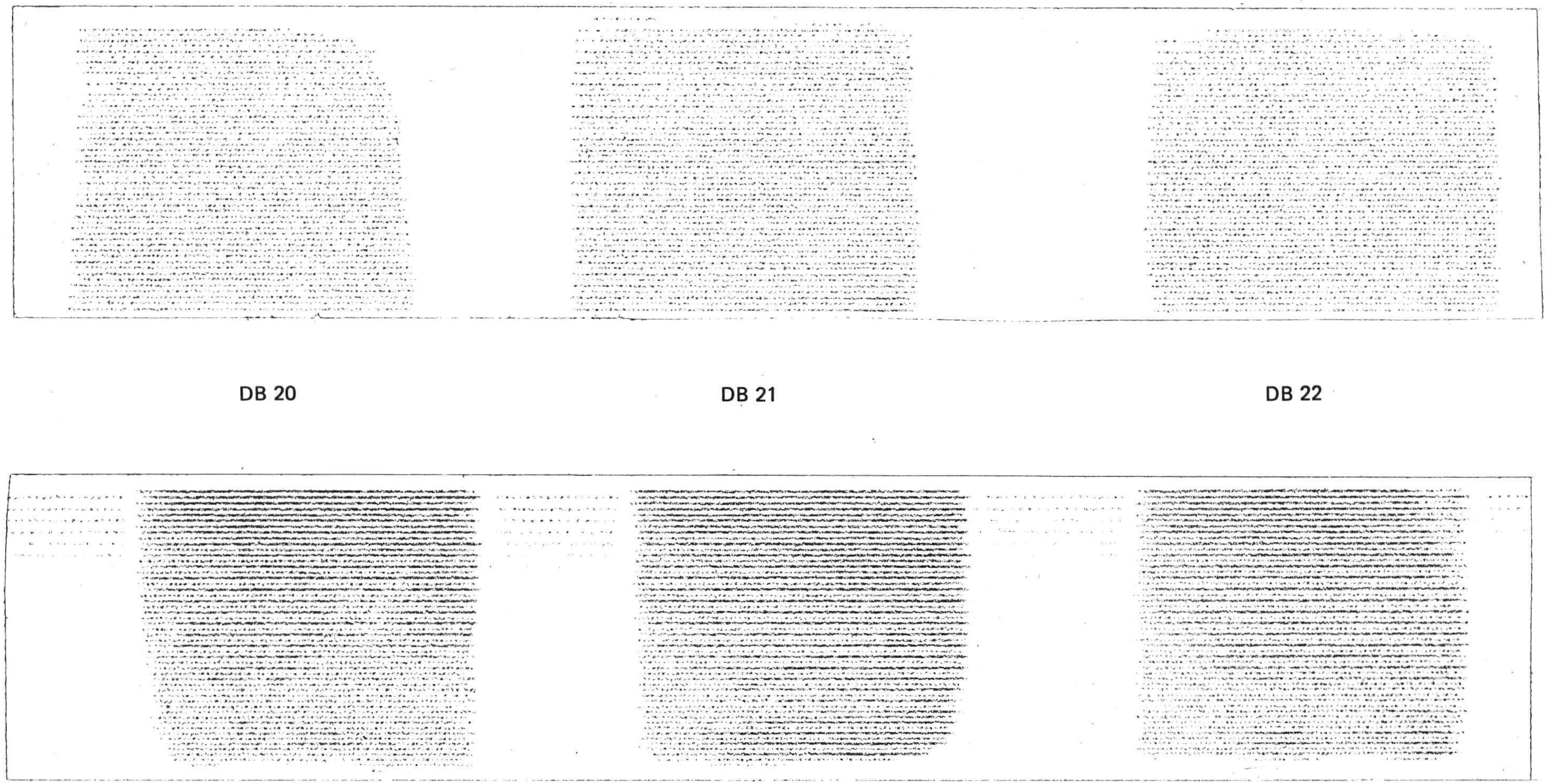


FIGURE 23 C SCAN RECORDING OF DIFFUSION BONDED B/AL PANELS .045 THICK, 7 PLYS

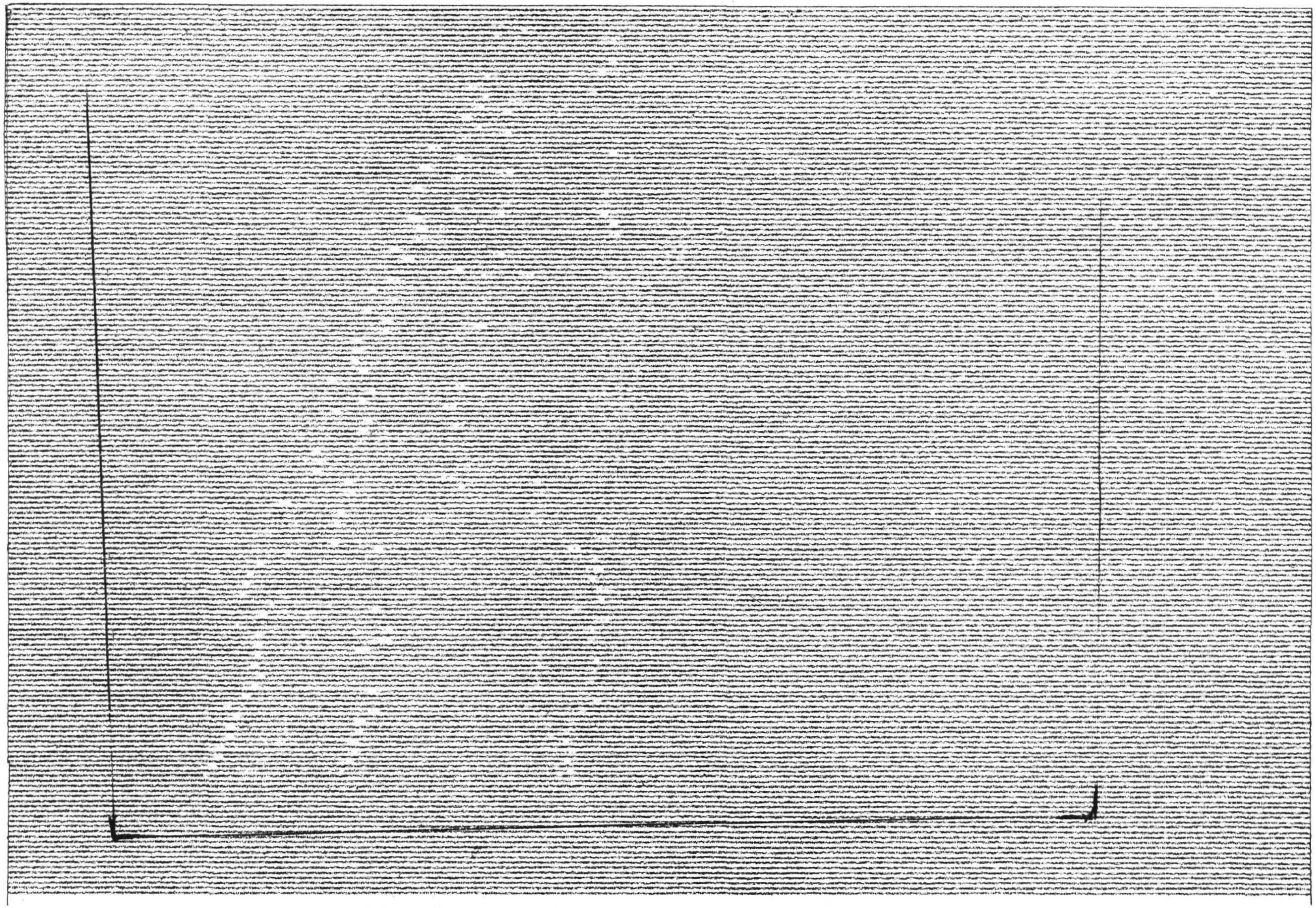


FIGURE 24 C SCAN RECORDING OF MONOLAYER ALUMINUM – STAINLESS STEEL MESH TAPE INDICATING GOOD QUALITY MATERIAL

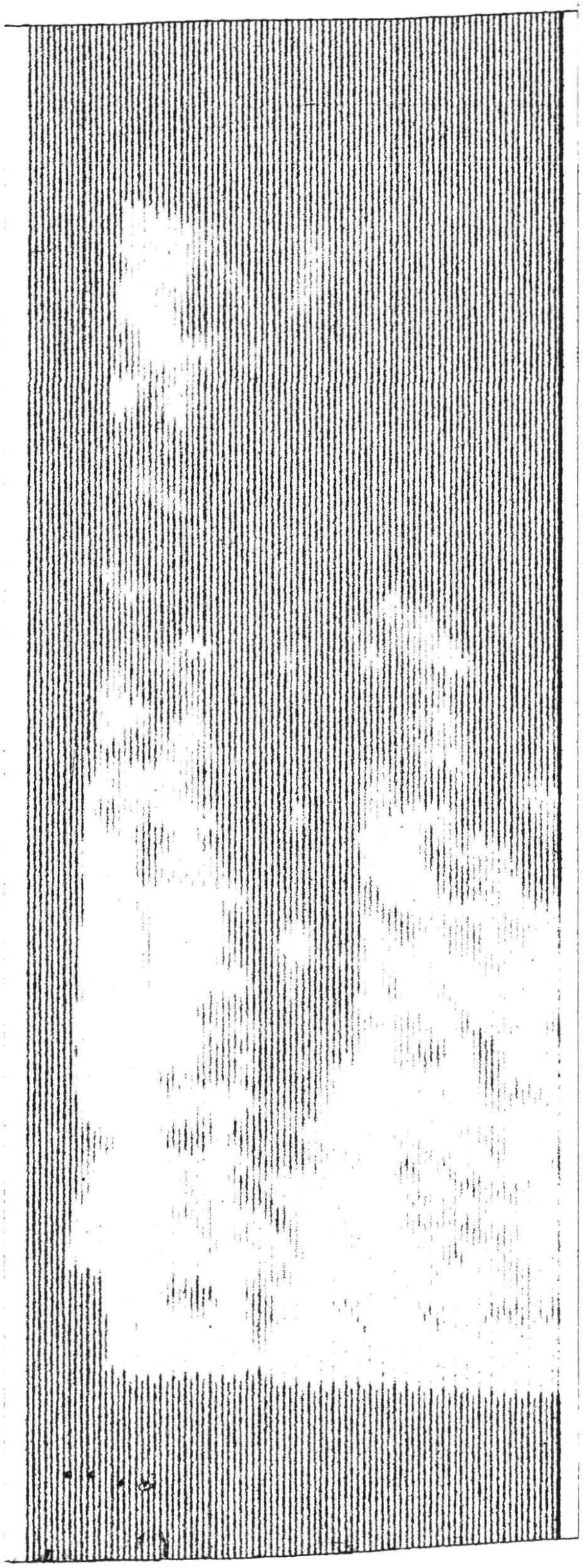


FIGURE 25 C-SCAN RECORDING INSPECTION OF DIFFUSION BONDED MONOLAYER STAINLESS STEEL TAPE SHOWING LARGE DISBOND AREAS

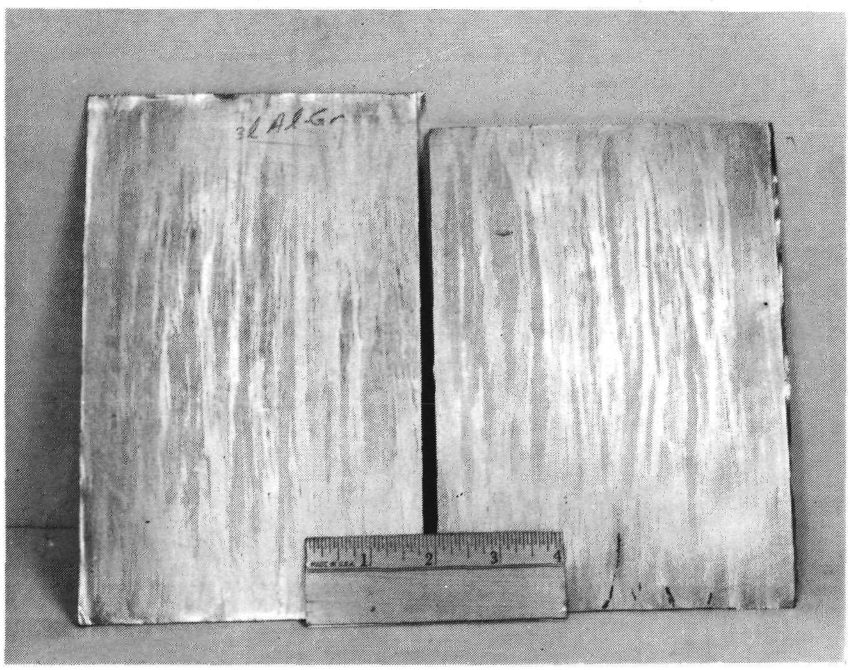


FIGURE 26 THREE PLY GRAPHITE/ALUMINUM PANELS DIFFUSION BONDED AT AMERCOM

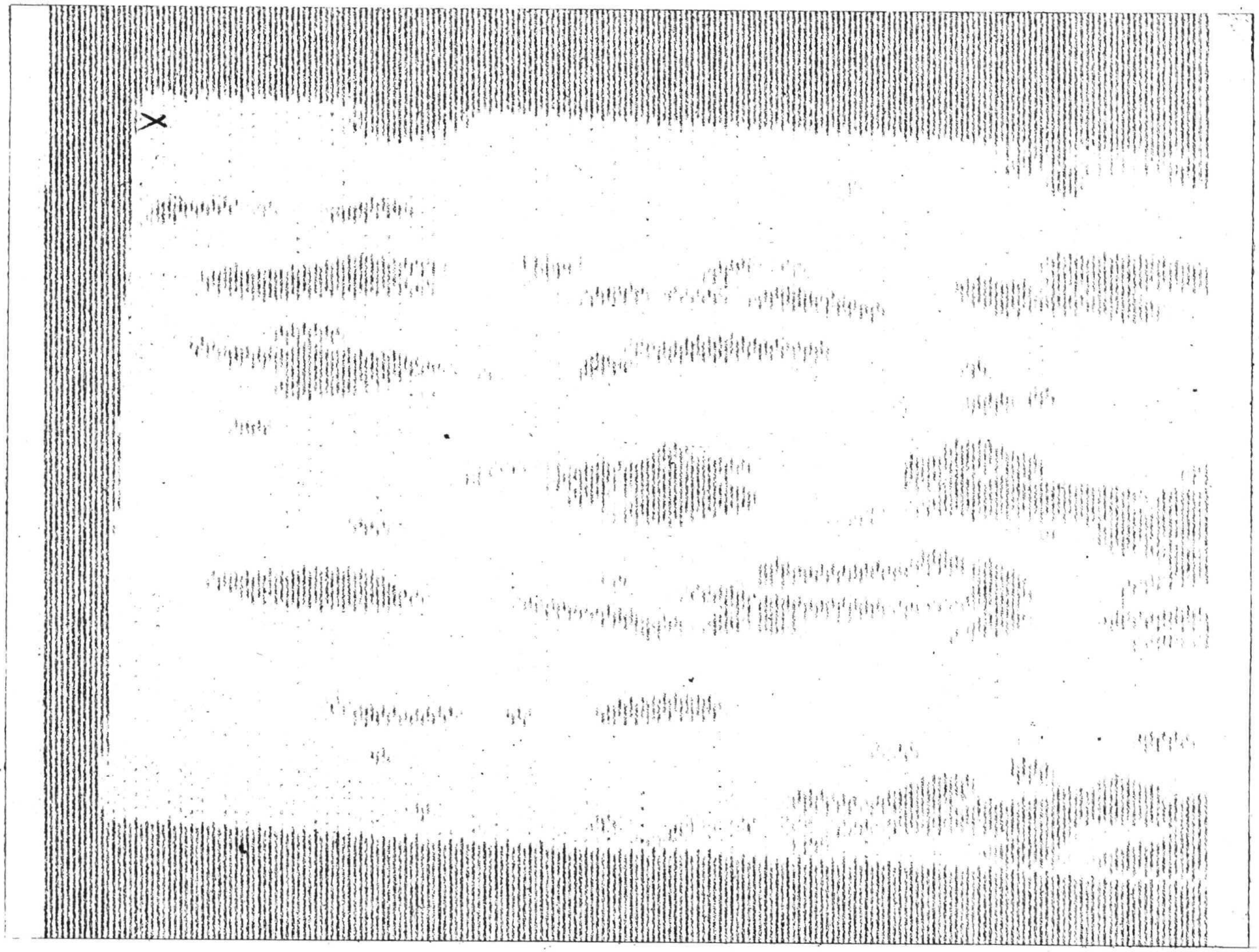


FIGURE 27 C SCAN RECORDING OF 3 PLY GRAPHITE/ALUMINUM LAYUP SHOWING VERY LITTLE BONDING

FIGURE 28 RADIOGRAPH OF 3 PLY GRAPHITE/ALUMINUM COMPOSITE PANEL

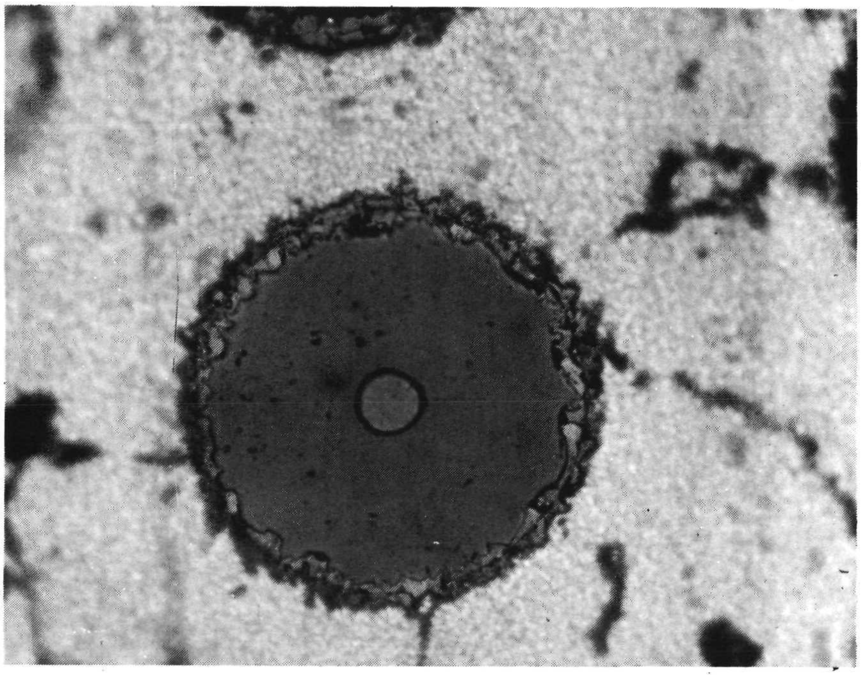
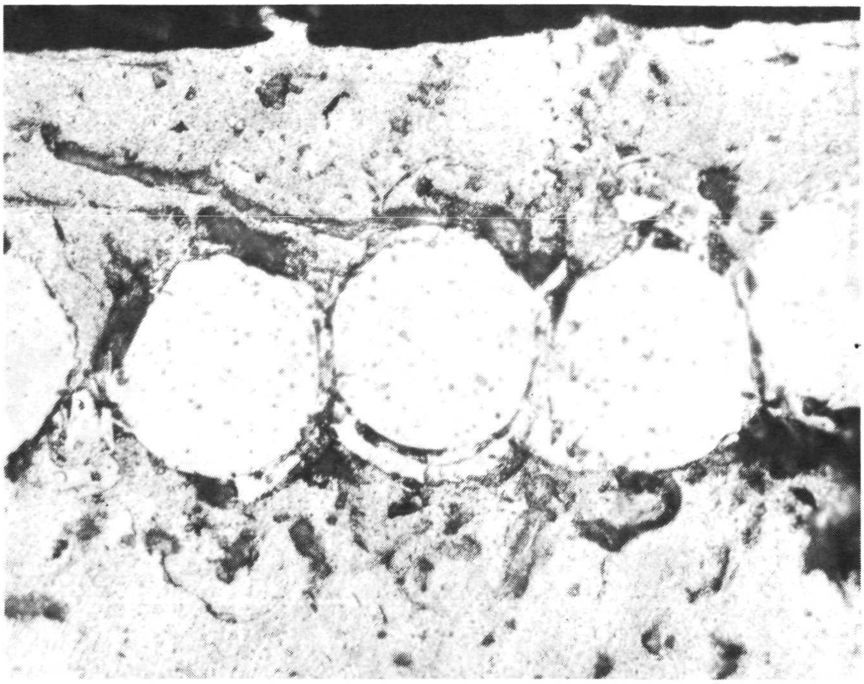
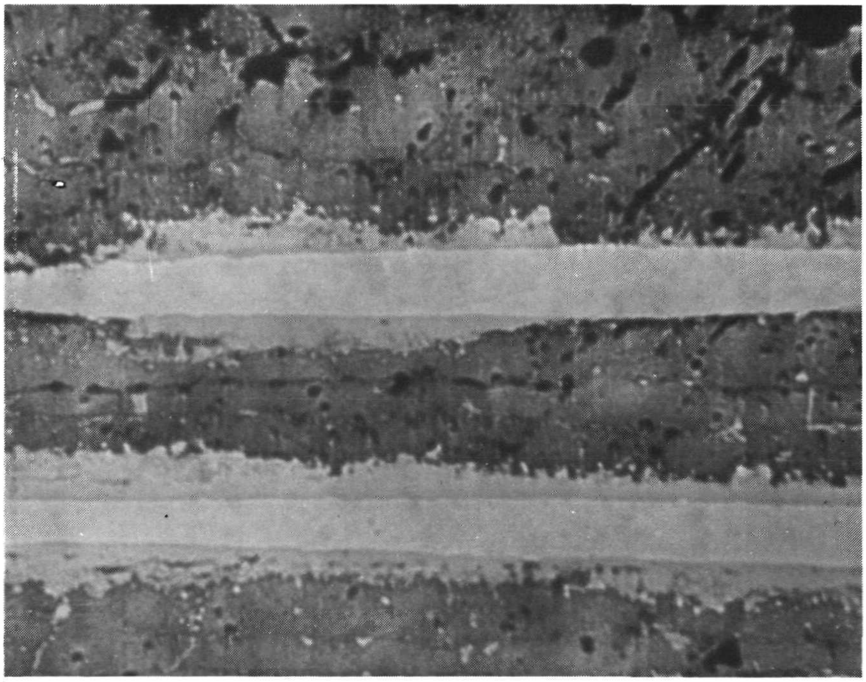

500X

FIGURE 29 ALUMINUM/BORON REACTION. REACTION PRODUCTS LAYER AT THE BORON-ALUMINUM INTERFACE



500X

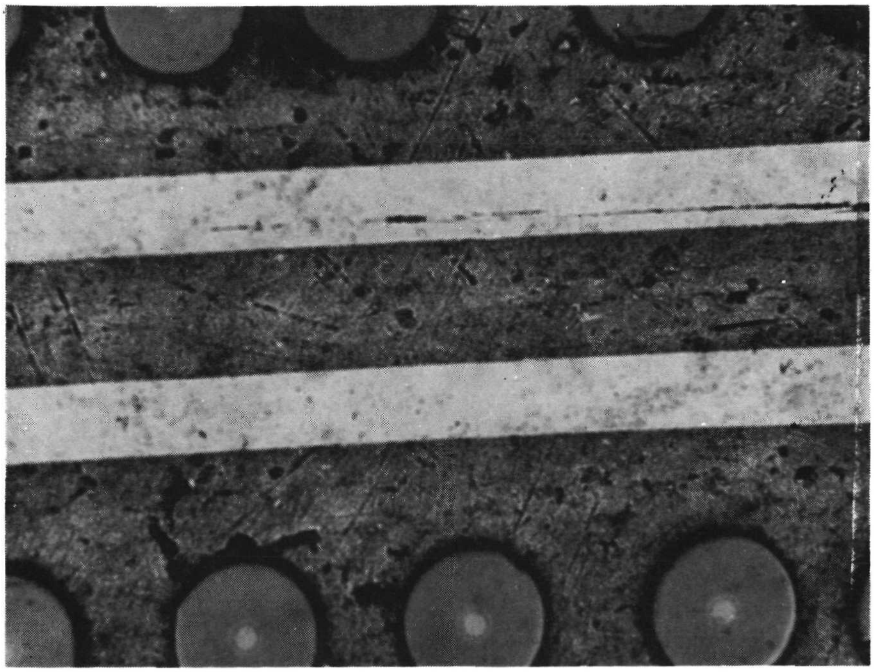
CROSS PLY END VIEW



500X

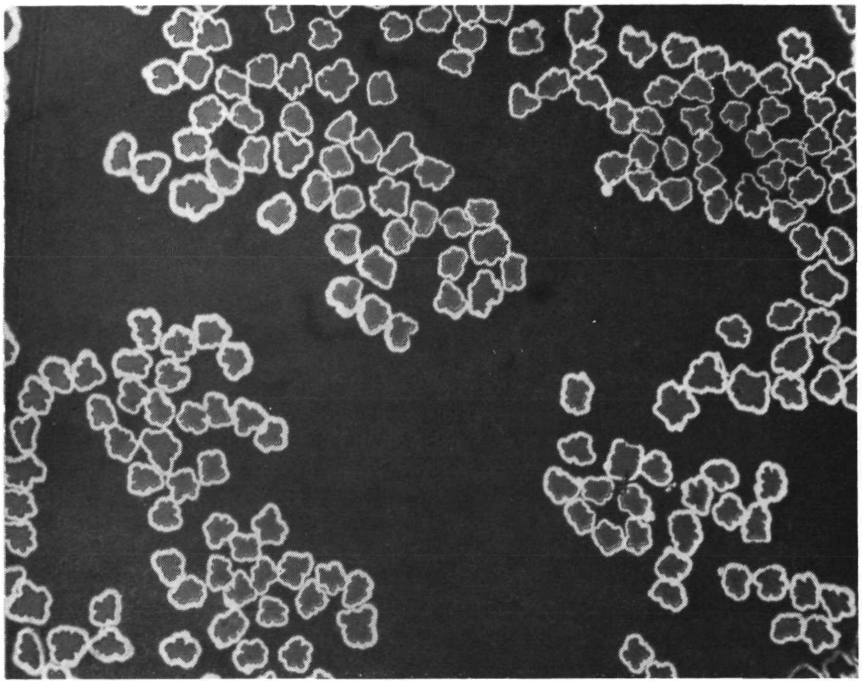
AXIAL VIEW

FIGURE 30 STAINLESS STEEL FILAMENT IN BORON/ALUMINUM COMPOSITE SHOWING INTERACTION PRODUCTS IN MATRIX



200X

FIGURE 31 TWO CROSS PLYS OF STAINLESS STEEL (AM350) FILAMENT TAPE DIFFUSION BONDED IN A BALANCED SIX PLY BORON/ALUMINUM 6061 MATRIX (30 – 35 V/O B) ~ AFTER THERMAL CYCLING



500X

FIGURE 32 NICKEL COATED THORNEL 50 GRAPHITE COATED BY VMSC

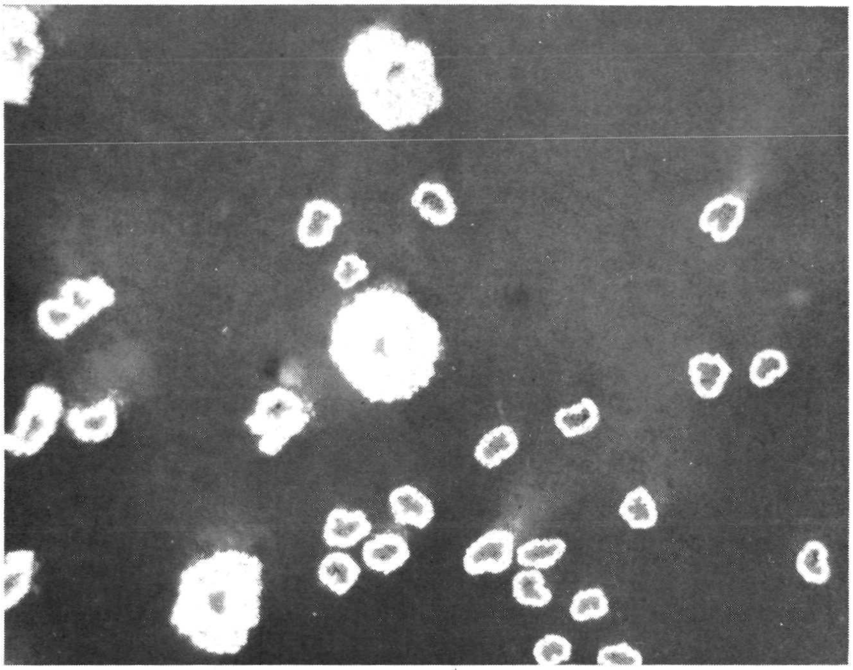
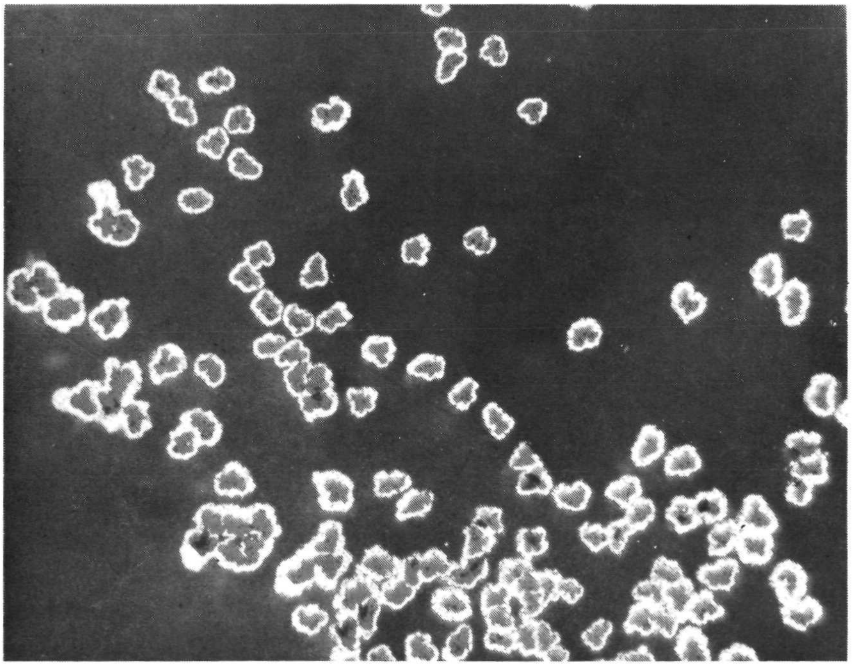
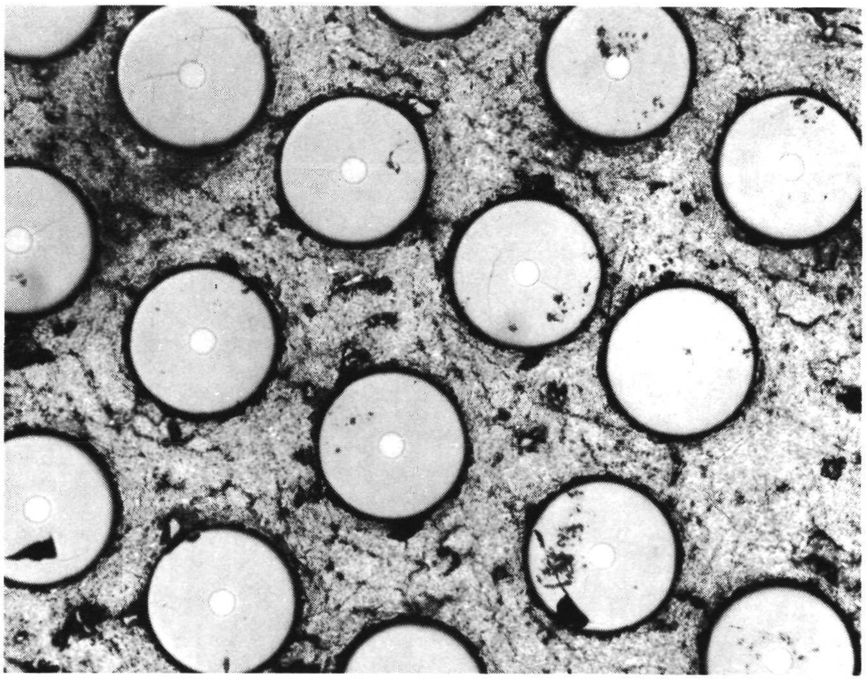


FIGURE 33 500X



500X

FIGURE 34 ALUMINUM COATED THORNEL 50 GRAPHITE



200X

FIGURE 35 DB–15 RUN BORON/ALUMINUM 7 PLY TAPE DIFFUSION BONDED

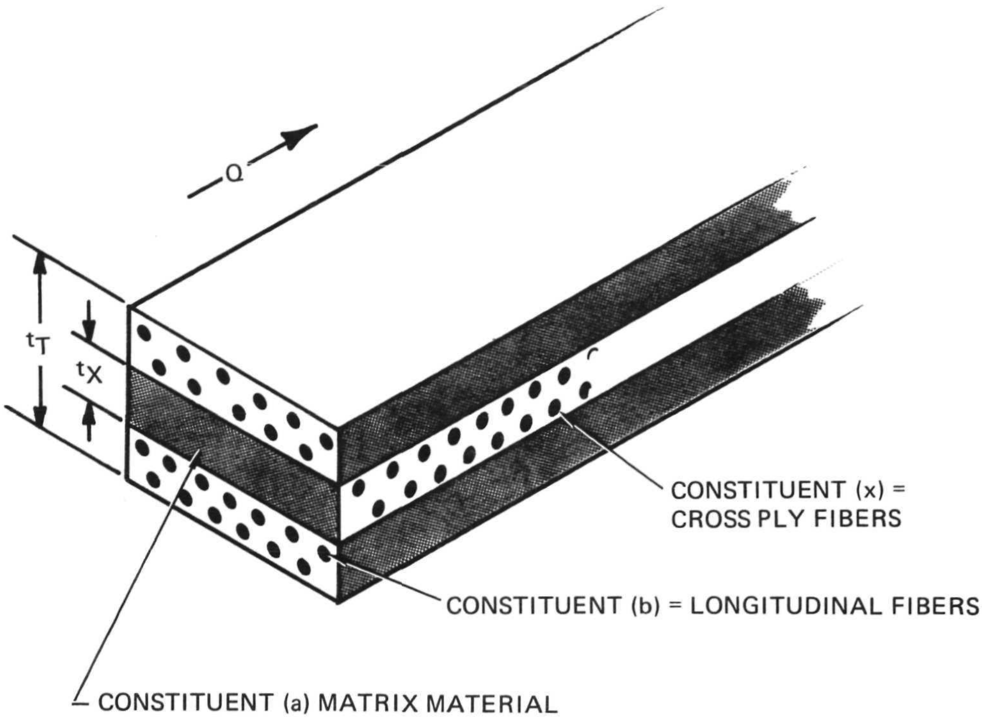


FIGURE 36 THERMAL CONDUCTIVITY MODEL

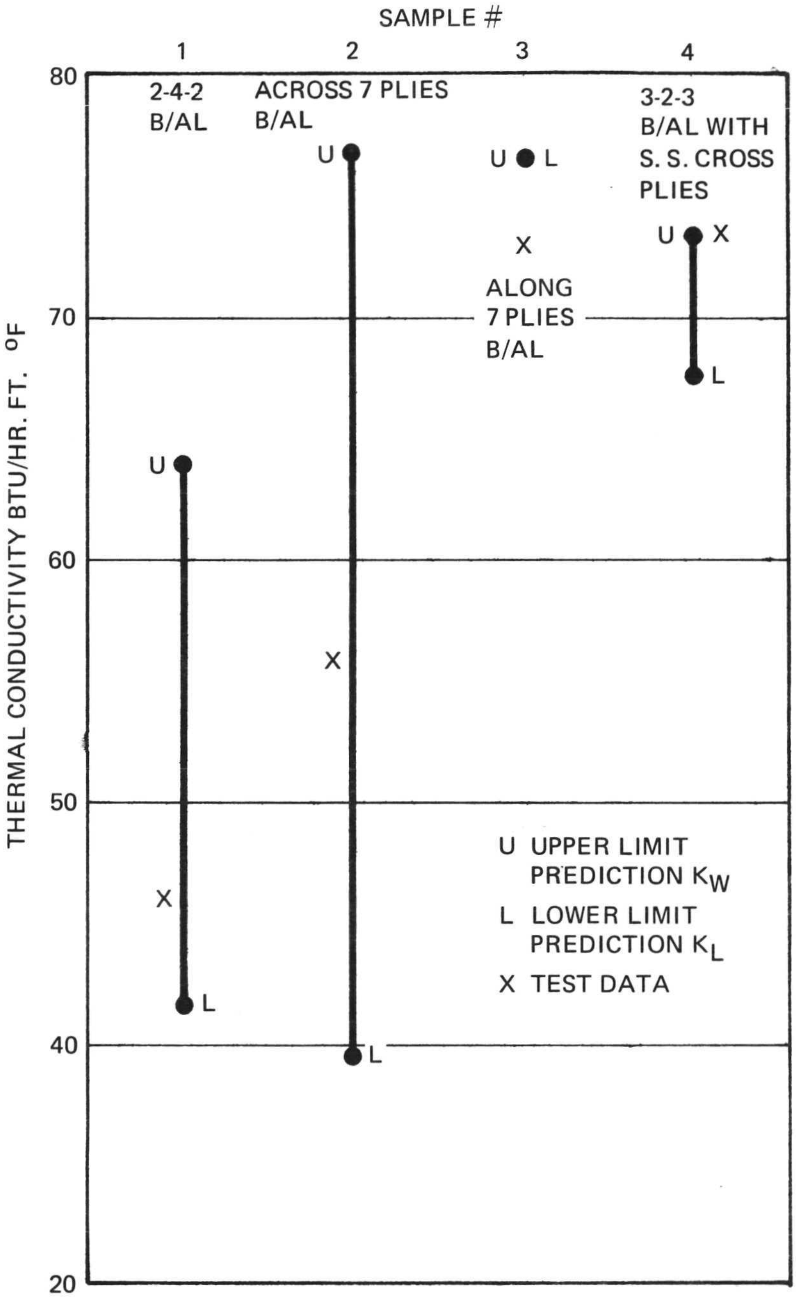


FIGURE 37 ROOM TEMPERATURE THERMAL CONDUCTIVITY

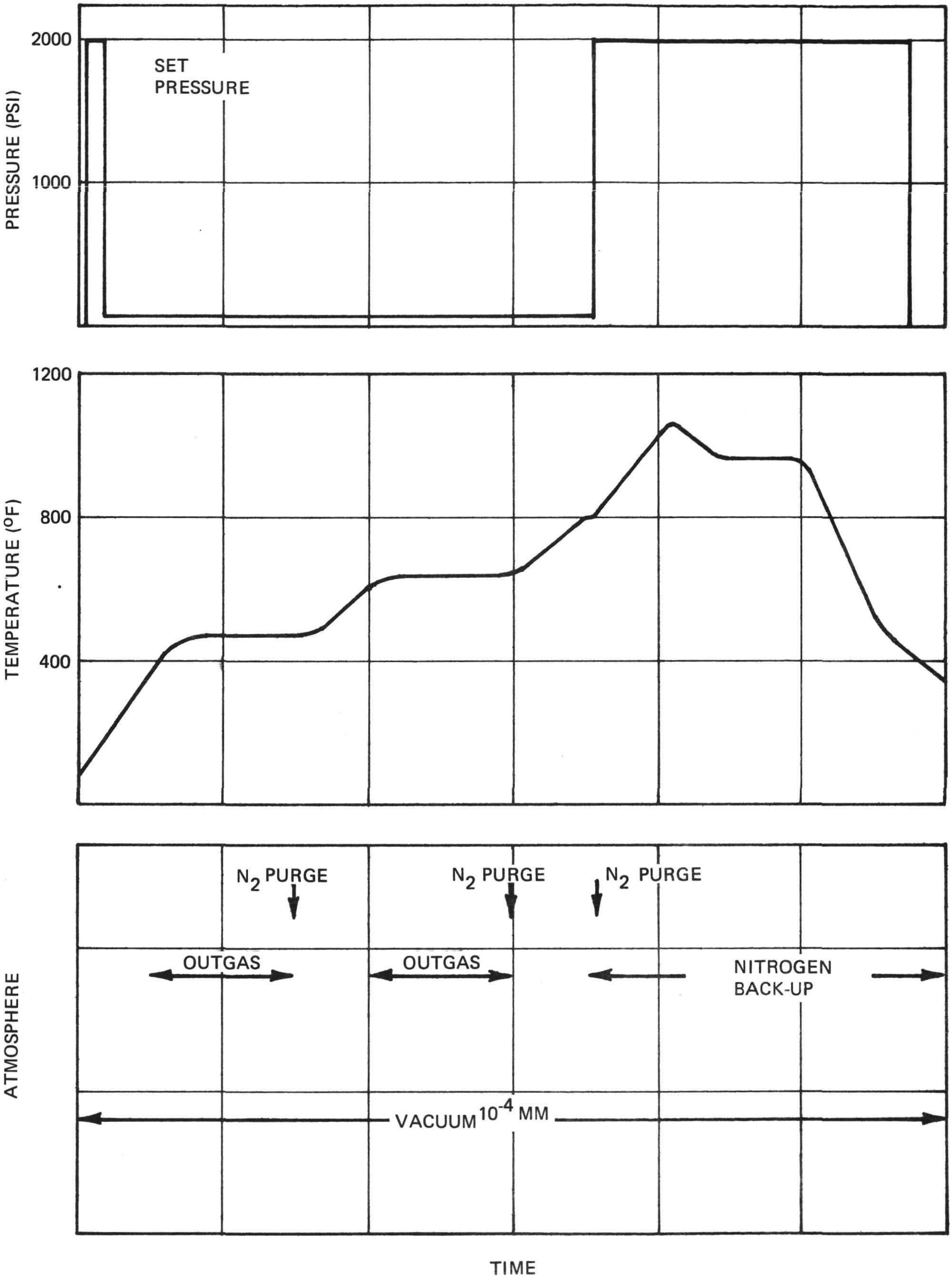


FIGURE 38 TYPICAL DIFFUSION BONDING CYCLE – HIGH PRESSURE

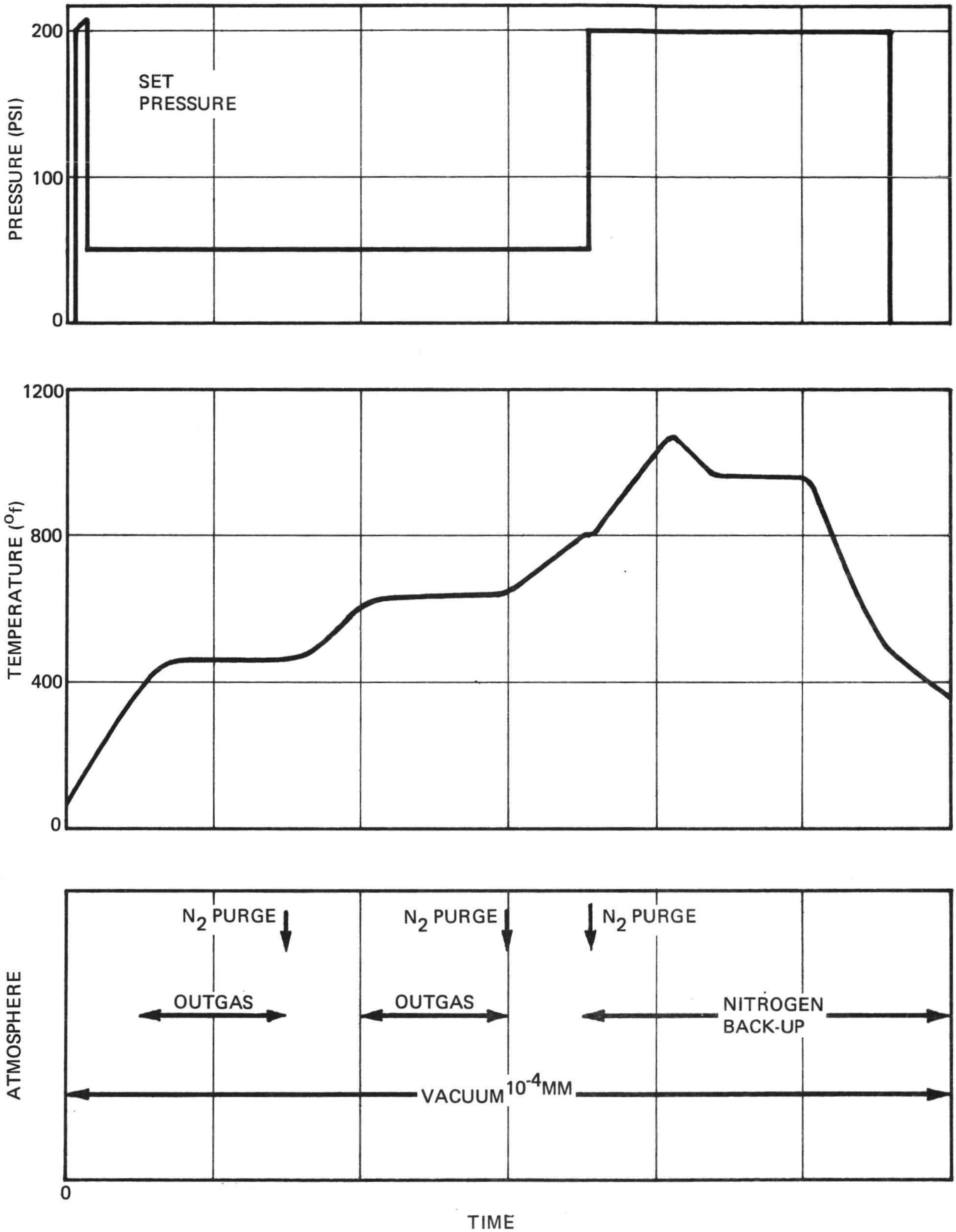


FIGURE 39 TYPICAL DIFFUSION BONDING CYCLE – LOW PRESSURE (AUTOCLAVE SIMULATION)

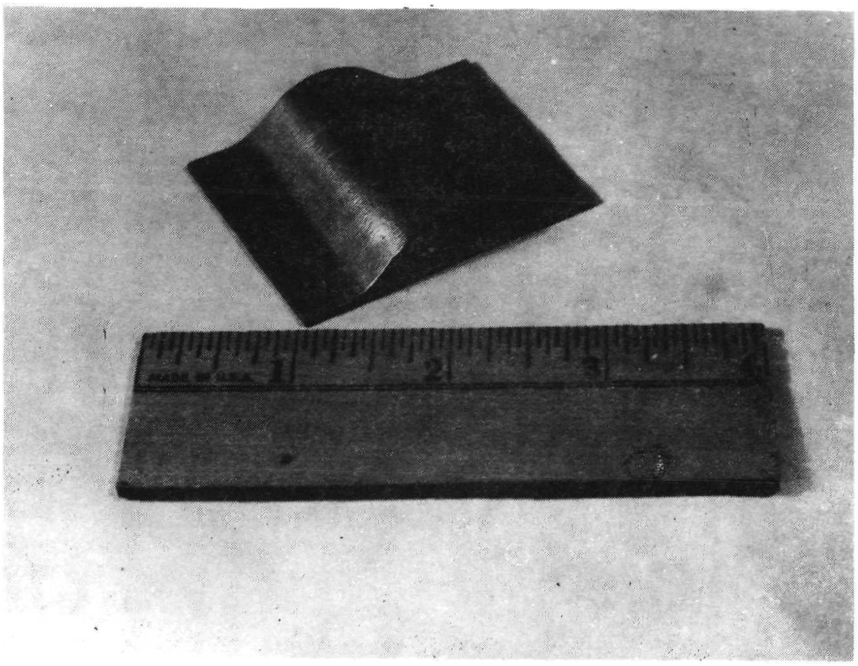
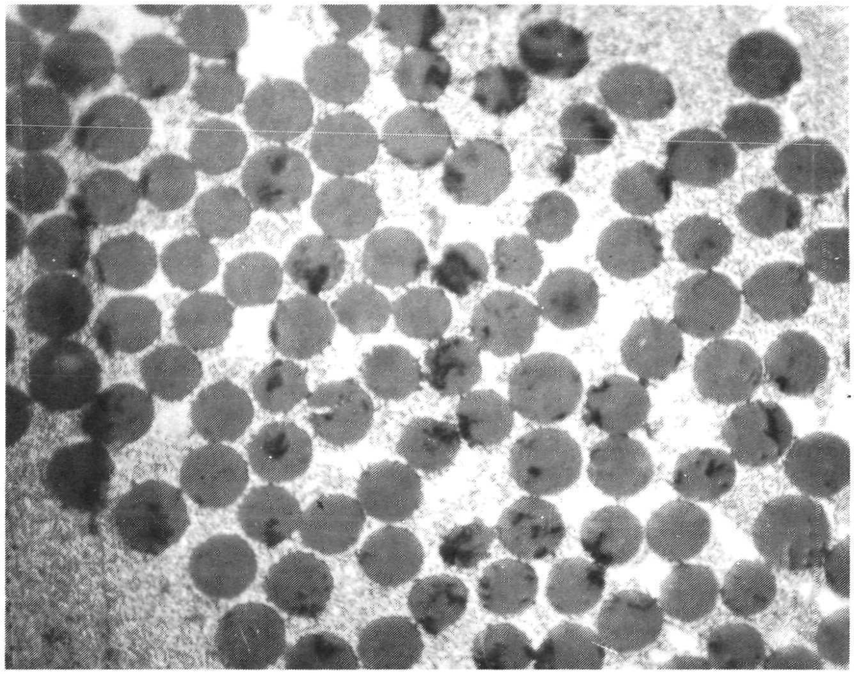
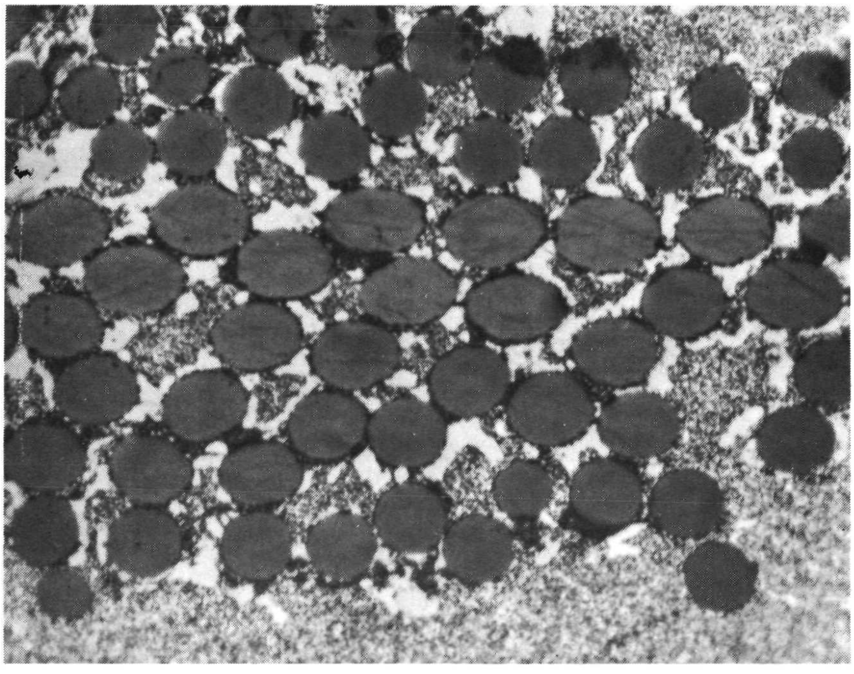


FIGURE 40 TWO PART DIFFUSION BONDED PANELS



1000X



1000X

FIGURE 41 GRAPHITE FILAMENTS DIFFUSION BONDED IN ALUMINUM MATRIX. NICKEL COATING ON FILAMENTS HAS UNDERGONE EXTENSIVE CHANGE. NOTE WHITE AREAS IN BOTTOM PHOTOGRAPH.

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